

# Impact of pixel detectors on SR experiments



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Brookhaven National Laboratory

# Outline

- SR Culture
- What is SR?
- Statement of problem
- Examples
- Summary

# Culture

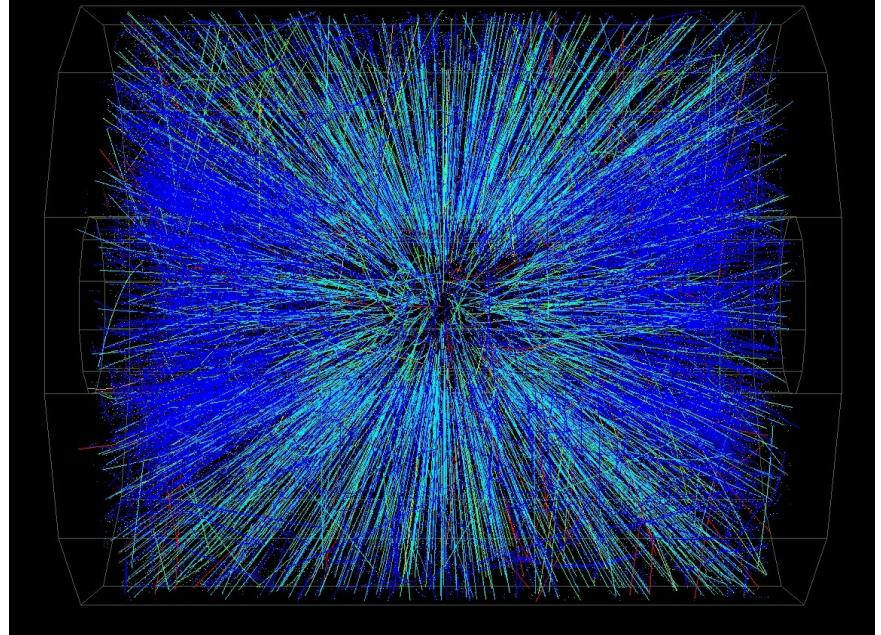
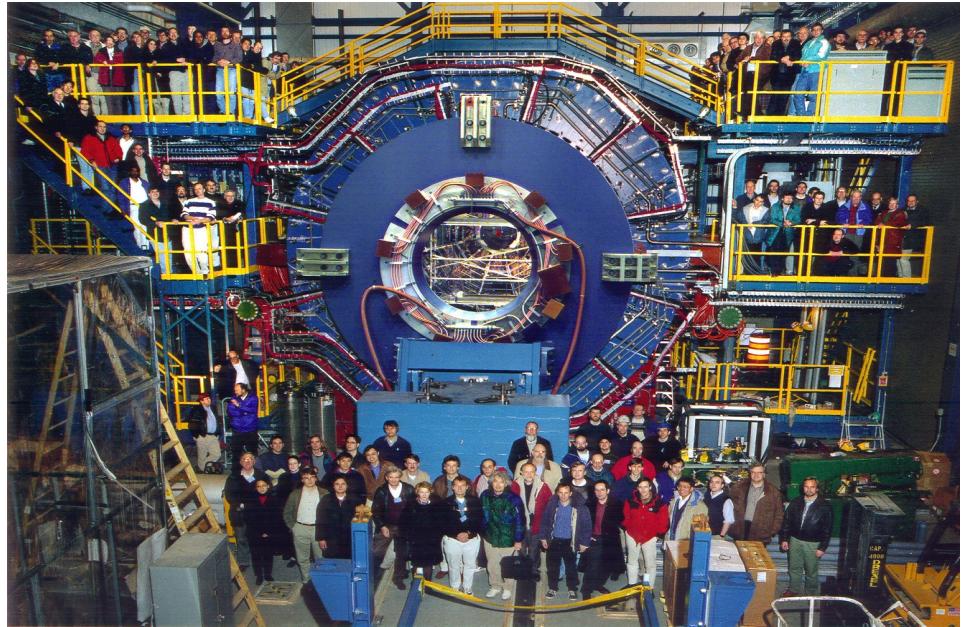
- SR and HEP are cultural opposites

- HEP: teams of hundreds for one experiment, complex detector system
- SR: teams of <10 usually, simple apparatus.
- HEP: Experiment takes years
- SR: Experiment takes hours or days
- HEP: Detector IS experiment
  - Scientists closely involved in design
- SR: SAMPLE is experiment: SR and detector a necessary evil
  - Scientists just want the result

# Culture

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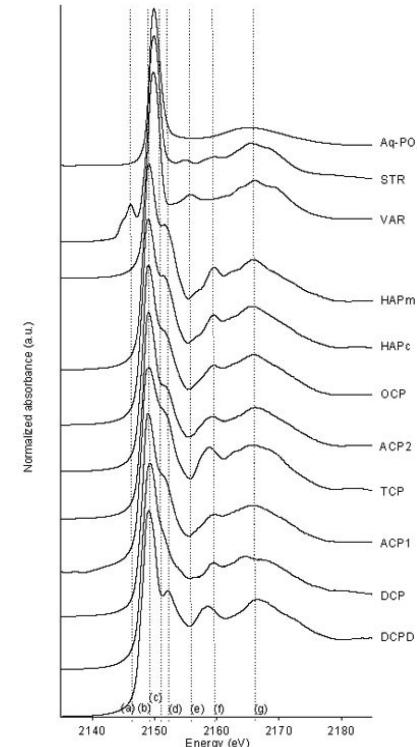
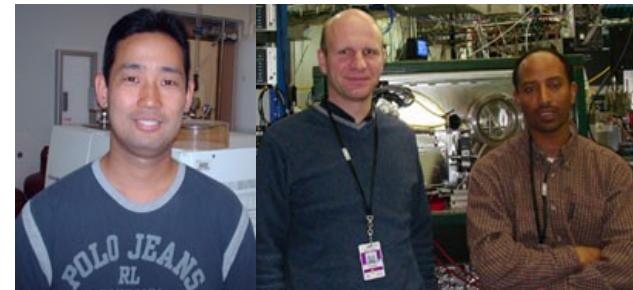
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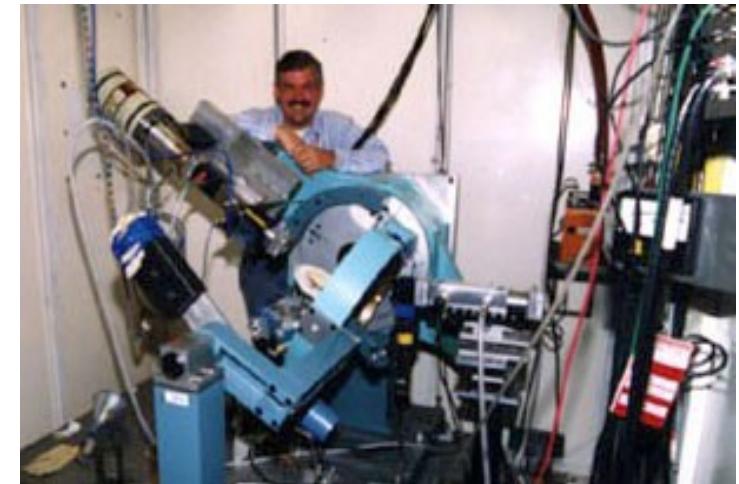
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# Synchrotron Radiation: the Photon Superprobe

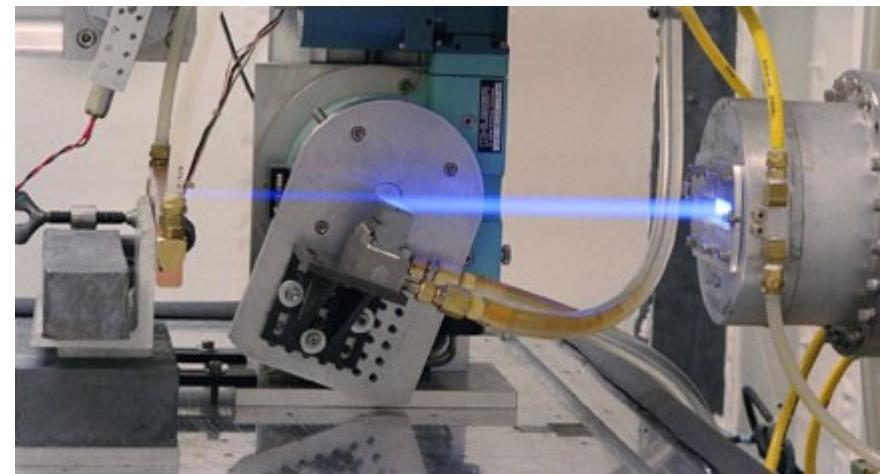
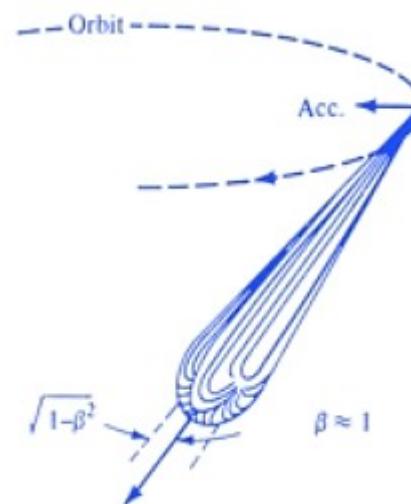
- Covers Infrared to Gamma-like energies:  $10^9$  range
  - Unique source in regions not covered by tunable lasers
- Different energy ranges need different instrumentation and different detector technologies
  - **IR**
  - **VUV**
  - **Soft X-ray**
  - **Hard X-ray**
  - **High-energy**



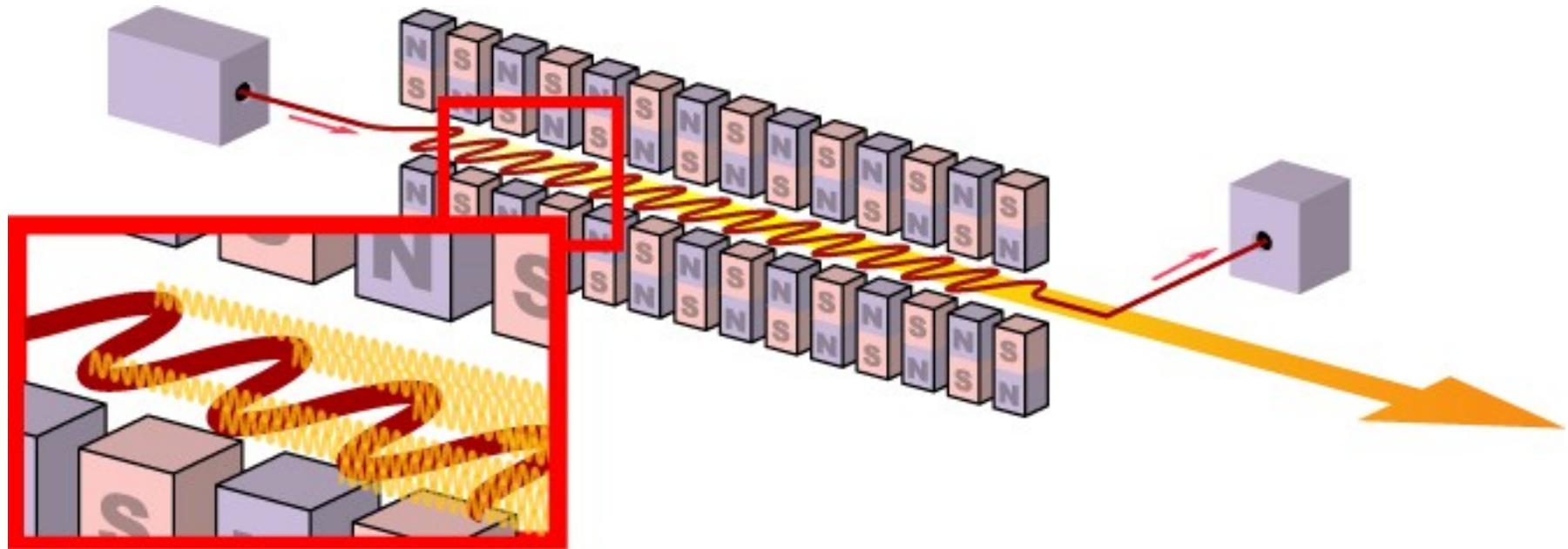
(From left) Peter Johnson (BNL), Tonica Valla (BNL), Zikri Yusof (University of Connecticut), Barry Wells (University of Connecticut).

# SR contd: Unique properties

- Very bright:
  - Very intense
  - Highly collimated
  - Large coherent fraction
- Polarized
  - spin-sensitivity
  - anisotropy sensitive
- Pulsed
  - time-resolved studies
- Has application in most scientific fields.



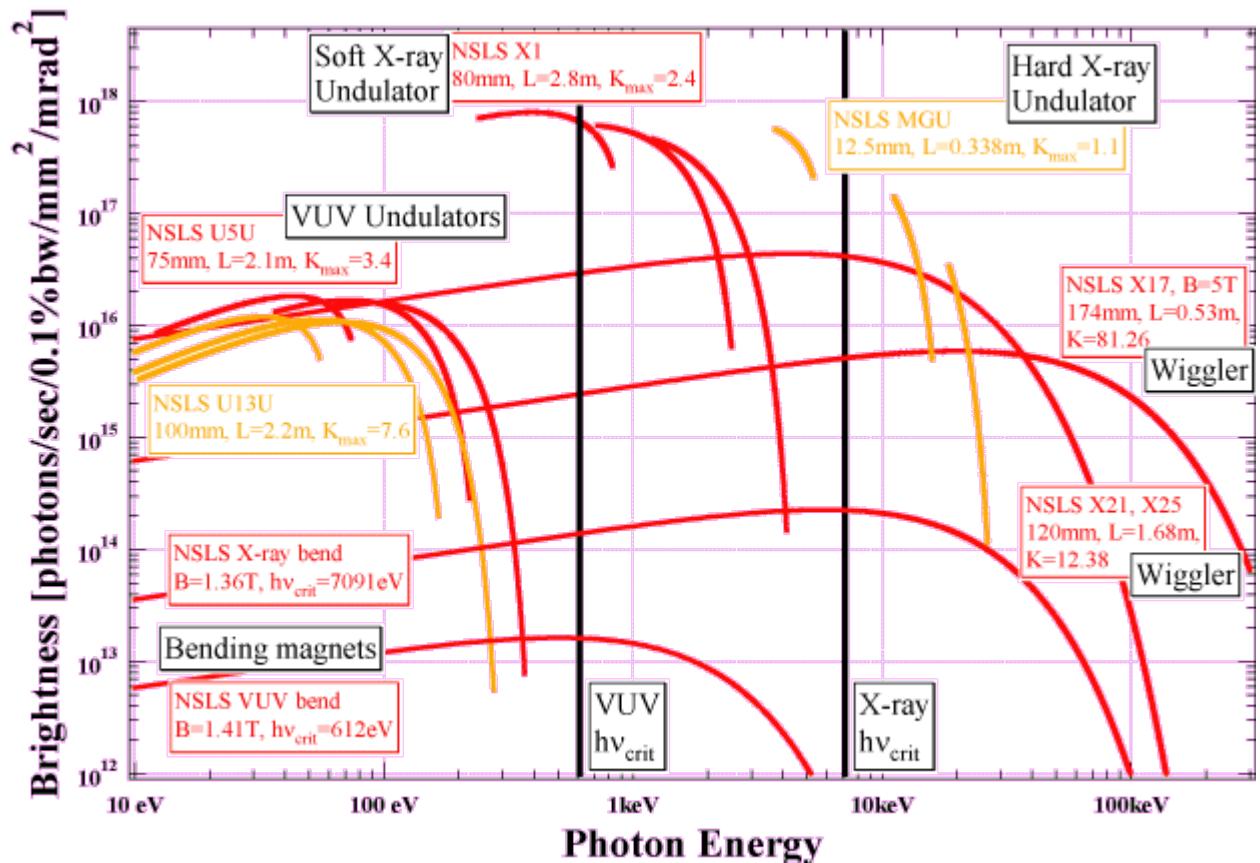
# Wiggles, Undulators and FELs



- Wiggler is series of strong bends alternating in sign
- Undulator is series of weak bends, so light emitted from successive bends has some coherence.
- FEL is very long undulator so radiation field is strong enough to introduce periodic microbunches inside bunch and hence a resonance with undulator.

# SR contd: Typical SR source spectra

- Wide variety of sources:
  - dipole magnets
  - wigglers
  - undulators
- Each have advantages and disadvantages



# SRSs worldwide



- 16 in USA
- 23 in Europe
- 25 in Asia
- 1 in Australia
- 1 in South America

# SRSs and FELs

- SRS is quasi-DC source (~10ns bunch spacing)
  - Electron or positron storage ring
  - No trigger, no 'free time' to dump data.
  - High average brightness, high stability
  - low peak brightness
  - fairly broadband source (~1% best case without filtering)
- FEL is pulsed source (~10ms bunch spacing)
  - Driven by LINAC / photocathode electron gun (low repetition rate)
  - Pulse width < 1ps
  - Low average brightness
  - Very high peak brightness
  - quasi-monochromatic ( $10^{-3}$  SASE,  $10^{-4}$  Seeded)

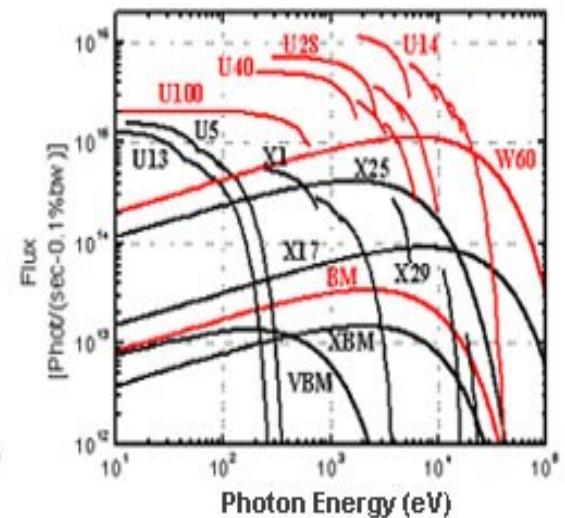
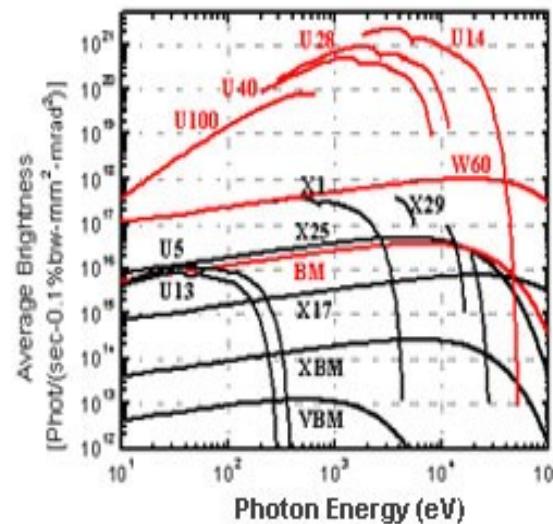
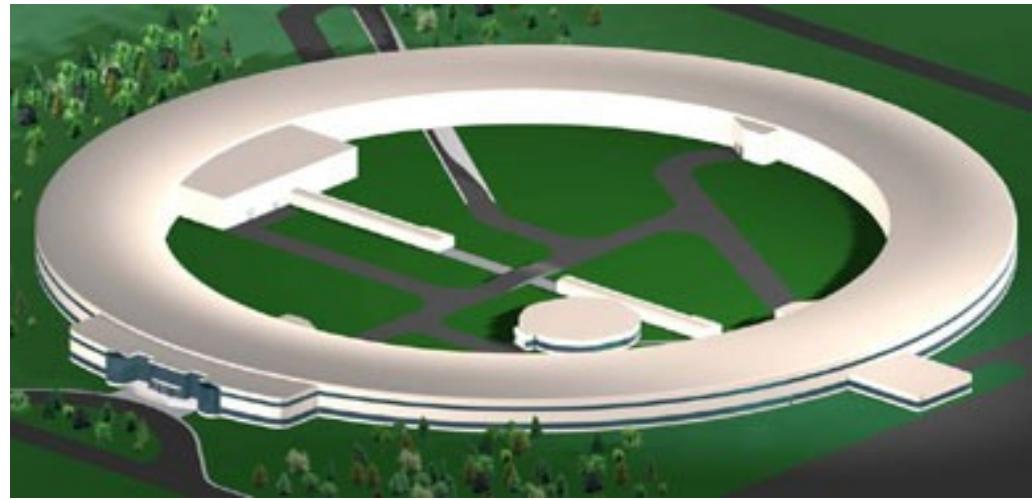
# Diamond Light Source (UK)

- Electron Beam Energy 3 GeV
- Circumference 561.6 m
- Number of cells 24 double-bend achromatic
- Straight sections 4 x 8 m, 18 x 5 m
- Beam current 300 mA (500 mA)
- Emittance 2.74 nm rad  
(horizontal) 0.0274 nm rad  
(vertical)
- Life time >10 h (20h)
- Max beamline length 40 m
- End-station capacity 30-40
- Phase I beamlines 7 for operation in January 2007



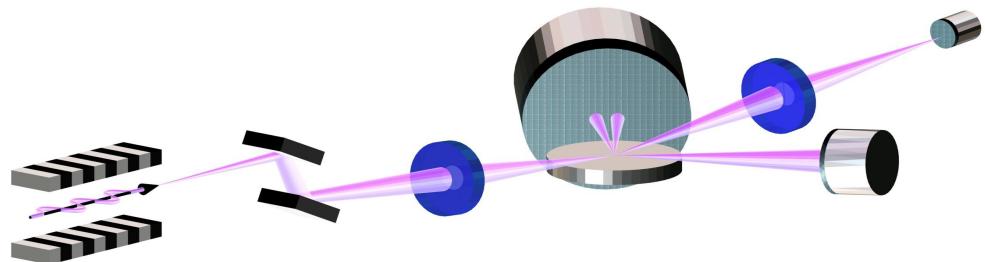
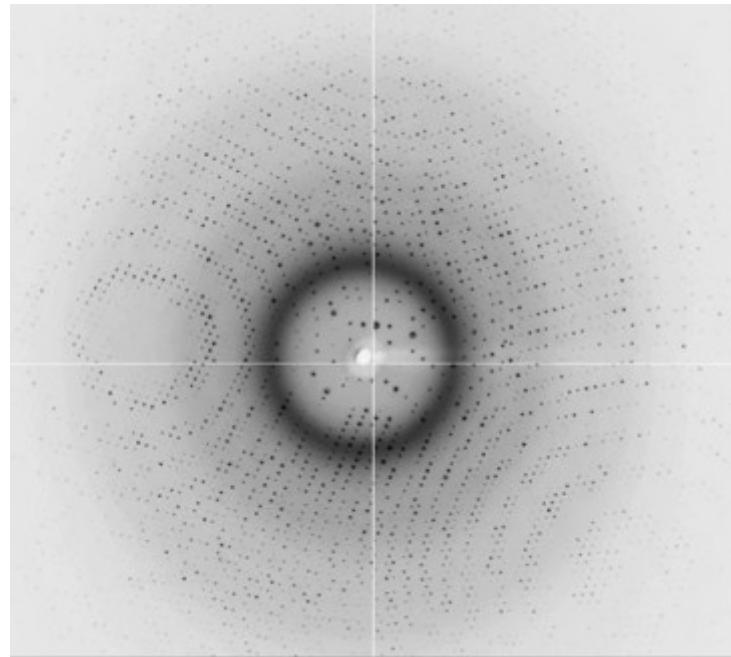
# NSLS-II

- A new 3rd-generation source at BNL
- 3GeV, 800m circumference.
- 30 DBA cells
- 6.6 & 8.6m straights
- <1nm-rad/0.008nm-rad
- Green-field site adjacent to NSLS
- 2014 ops.



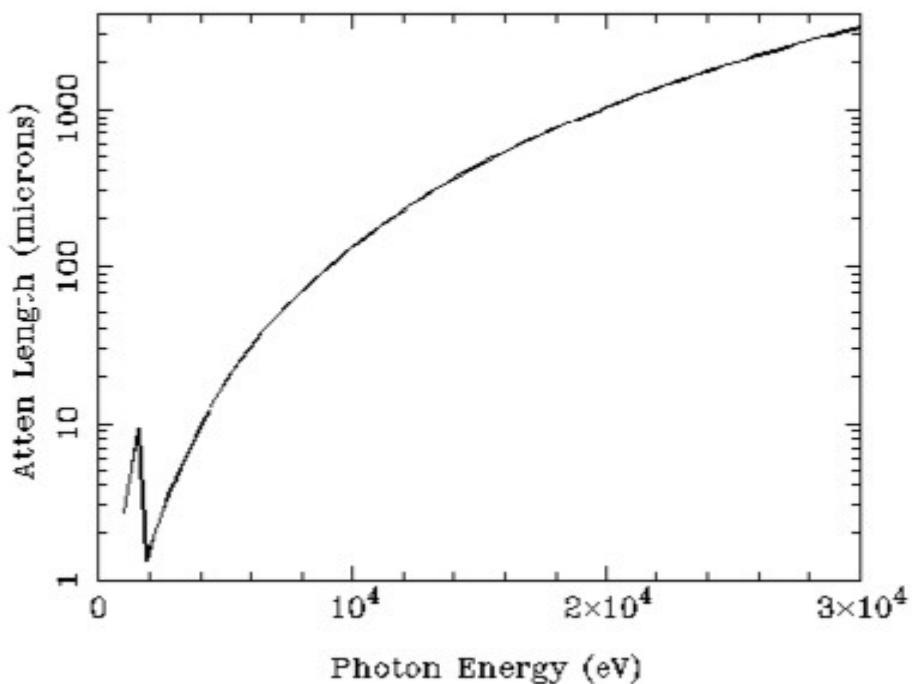
# Detector challenges: SR

- Dynamic range
  - Photon counting
    - Energy range
    - Rate
    - Energy resolution
- Coverage
  - Area & spatial resolution, Fast readout of 2D detectors
- Multi-dimensionality
  - Space, Energy, Time, Temp., Press.
- Multiple concurrent methodologies

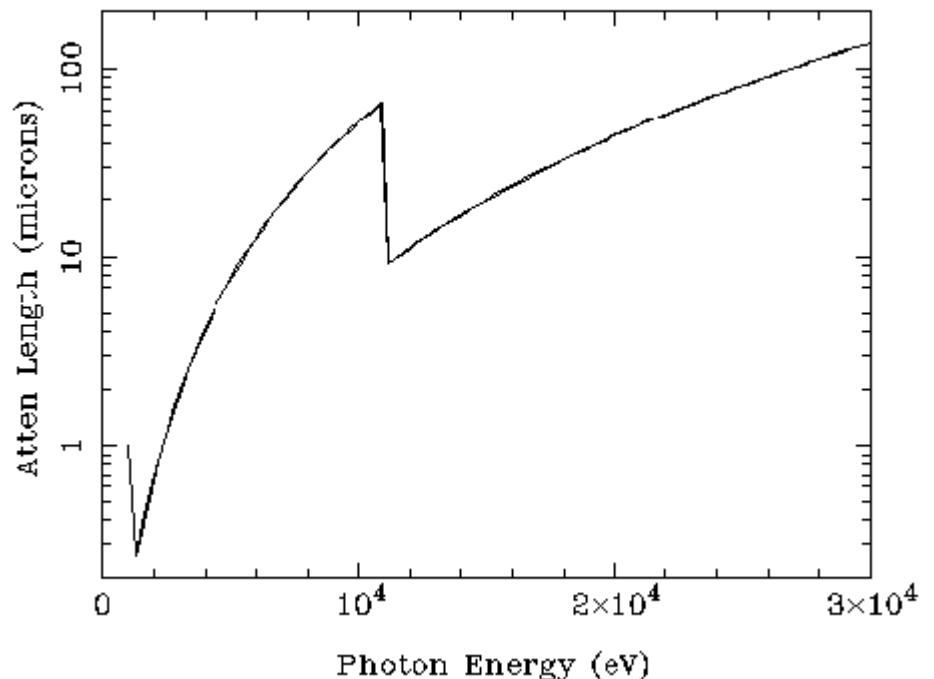


# Absorption length for Si & Ge

Si Density=2.33, Angle=90.deg



Ge Density=5.323, Angle=90.deg



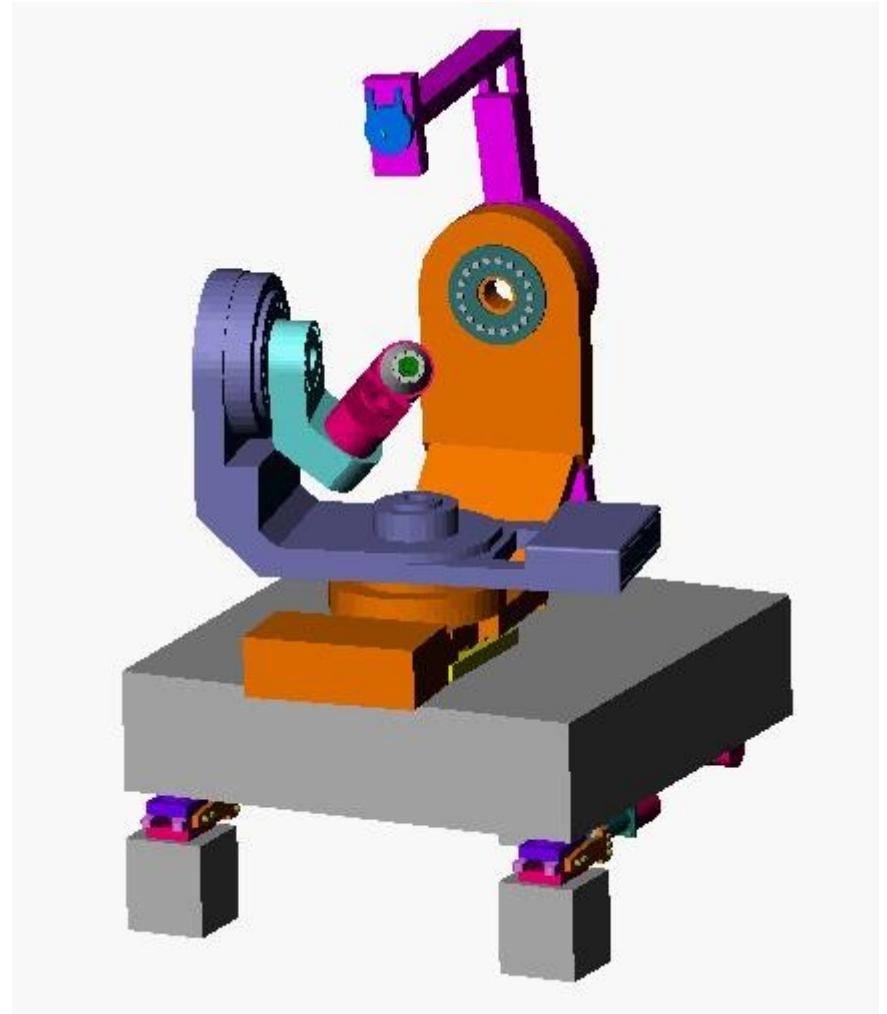
- Materials science needs  $E > 20\text{keV}$  to penetrate dense materials (alloys, ceramics etc.)
- Biology needs higher  $E$  to reduce radiation damage

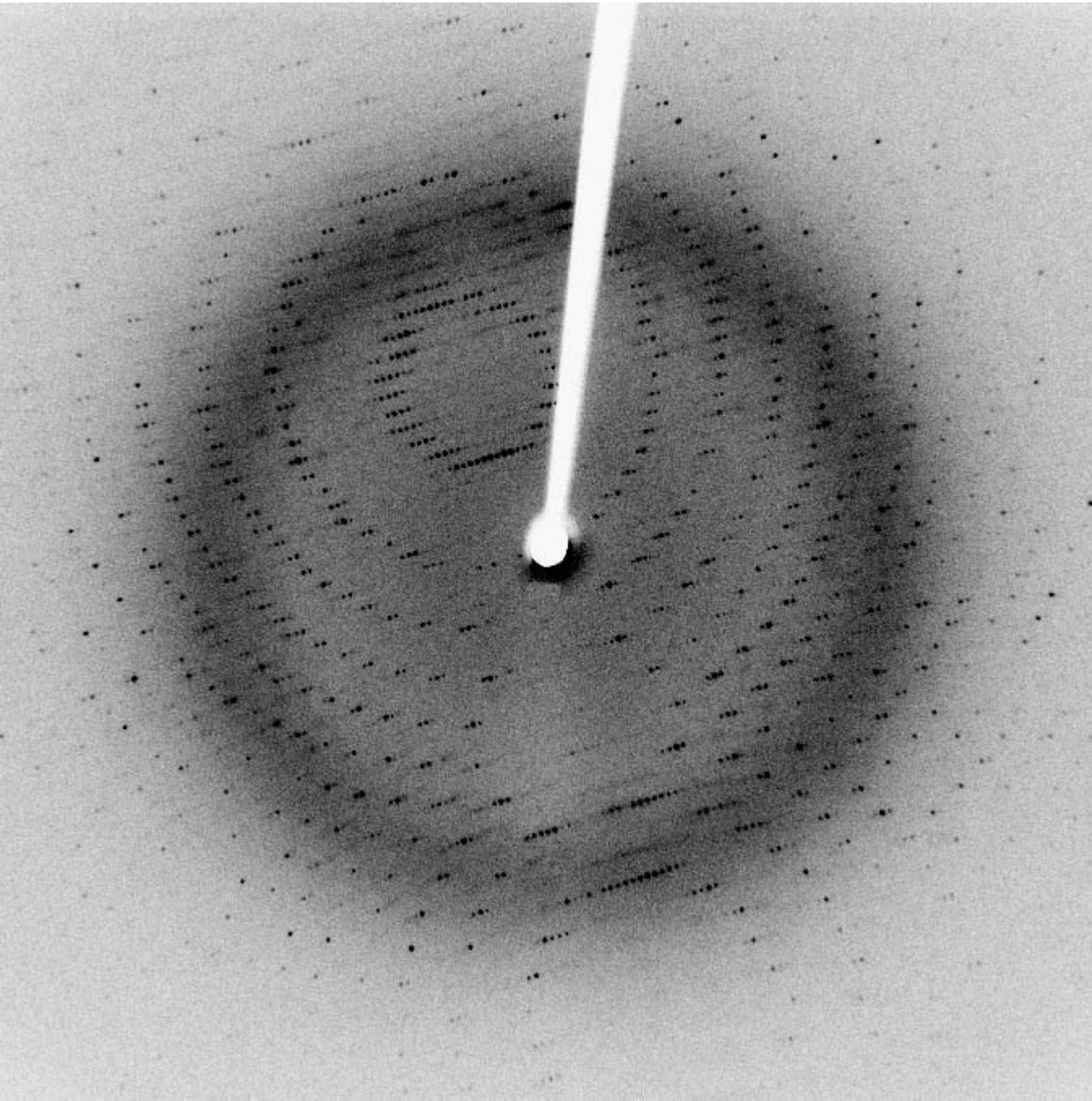
# SR X-ray techniques

- Scattering & diffraction
  - Crystallography
  - Small-angle scattering
  - Diffuse scattering
  - PDF
- Spectroscopy
  - Fluorescence
  - EXAFS & XANES
- Imaging & microscopy
  - Scanning probe microscope
  - Full-field microscope
  - Coherent diffraction & Holography

# Crystallography: Sample MUST move

- Complex goniometry
  - to allow sample to have an arbitrary orientation w.r.t. the incident x-ray beam, with minimum blind regions.





**NSLS**  
NATIONAL SYNCHROTRON LIGHT SOURCE

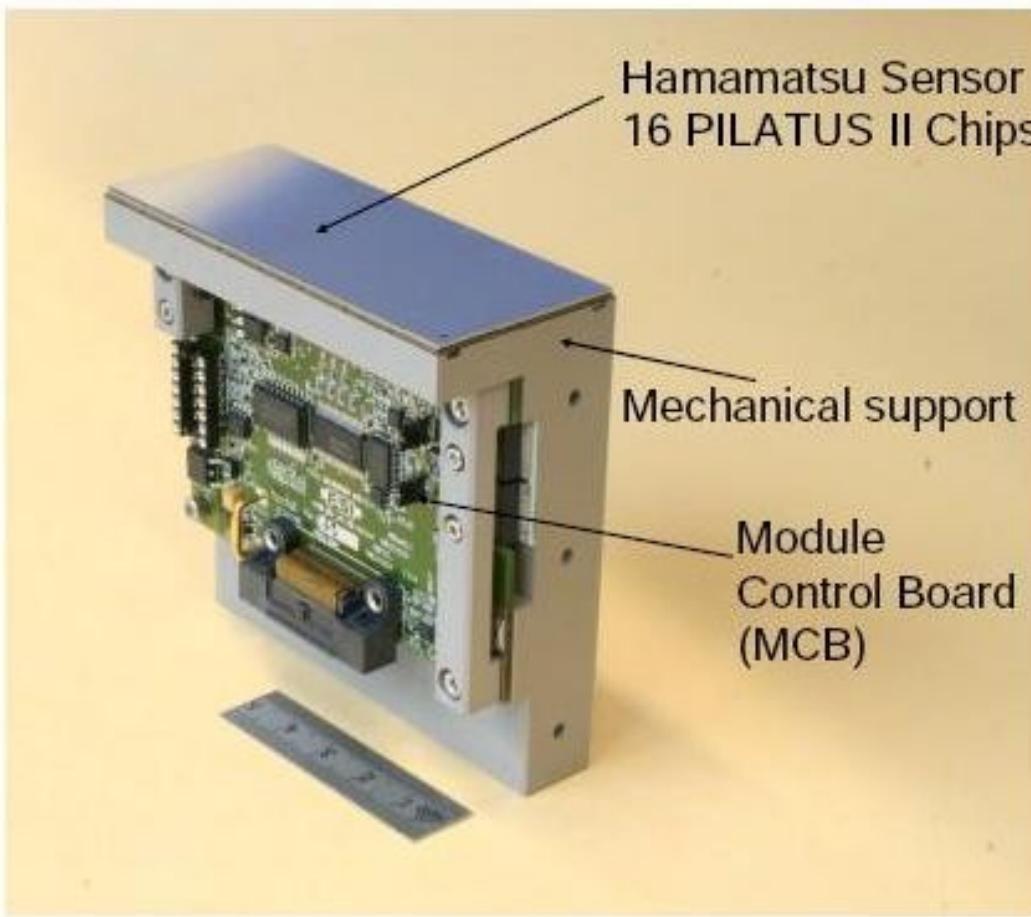


**BROOKHAVEN**  
NATIONAL LABORATORY

*Instrumentation Division*



## Pilatus Module



- 1 Silicon Sensor
- 16 PILATUS CMOS Chips
- 487 x 195 pixels = 94965 pixels
- Active Area 83.8 x 33.6 mm<sup>2</sup>
- $T_{ro} = 3.6 \text{ ms}$
- Continuously sensitive: no gaps between chips
- Building Block of all Pilatus Detectors

# Large area detectors

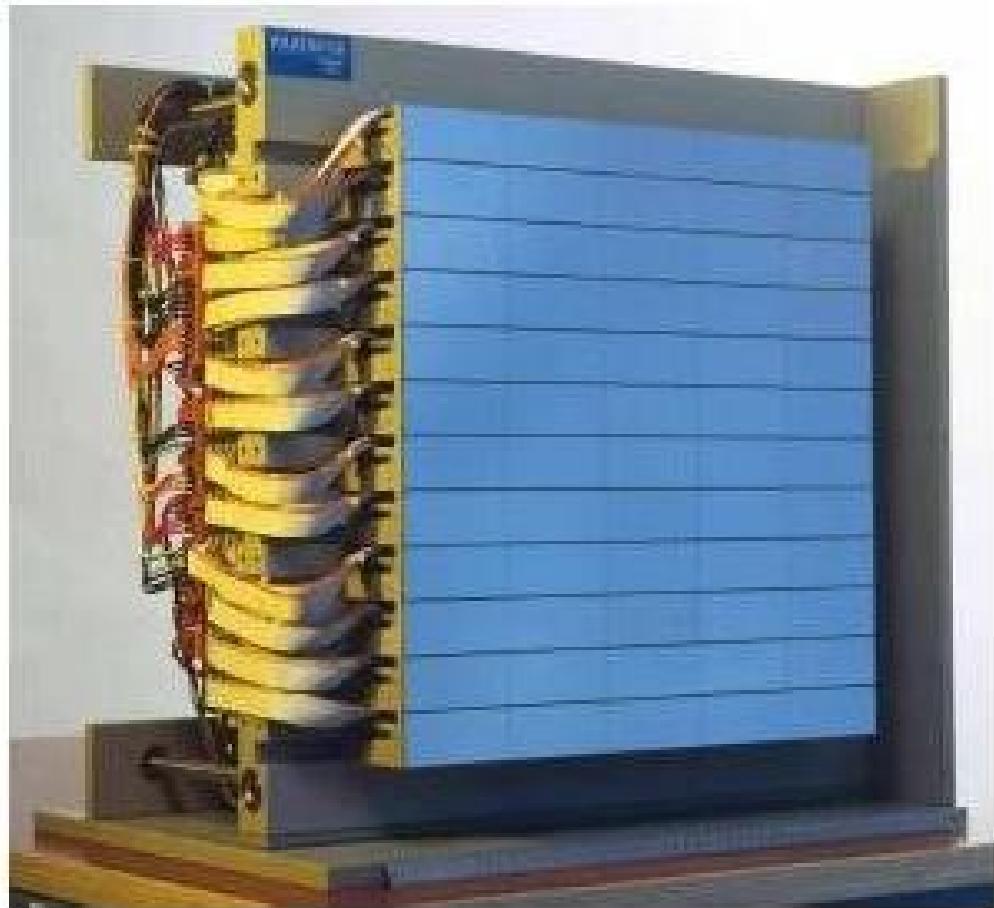


Figure 5: The different multimodule systems. Left: PILATUS 6M (5x12 modules). Right: PILATUS 2M (3x8 modules).

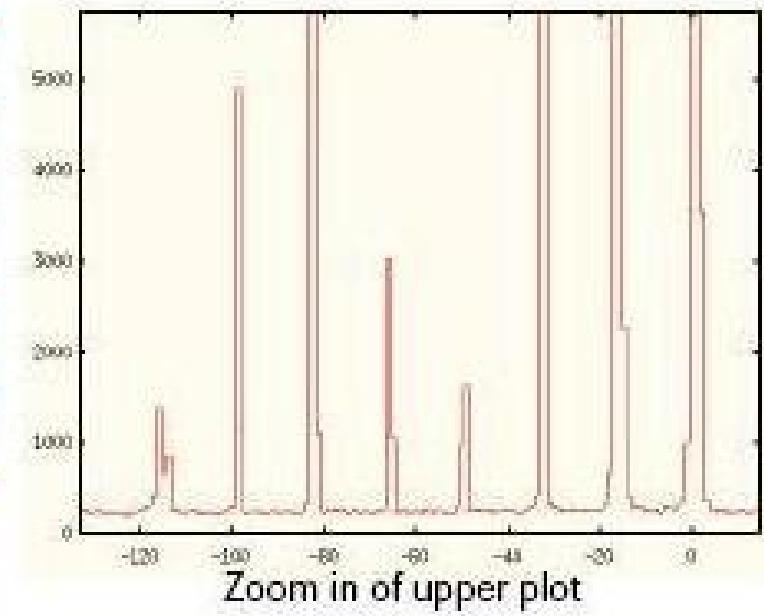
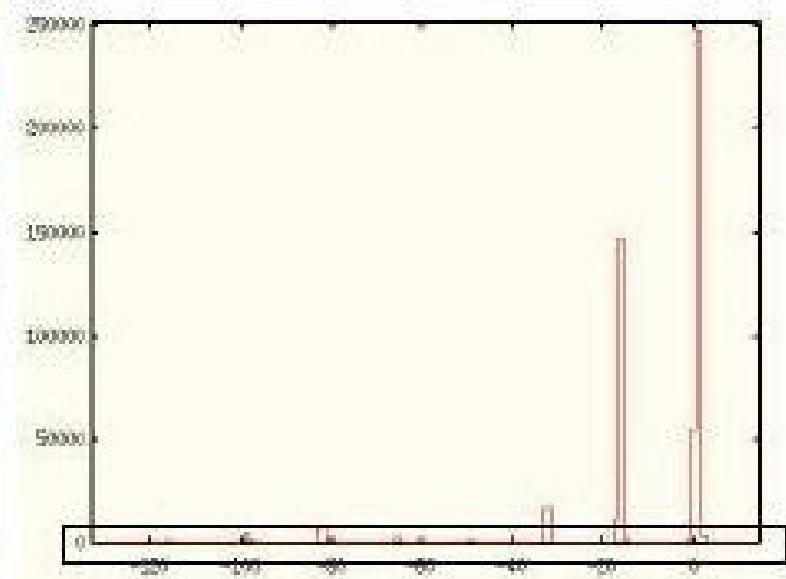
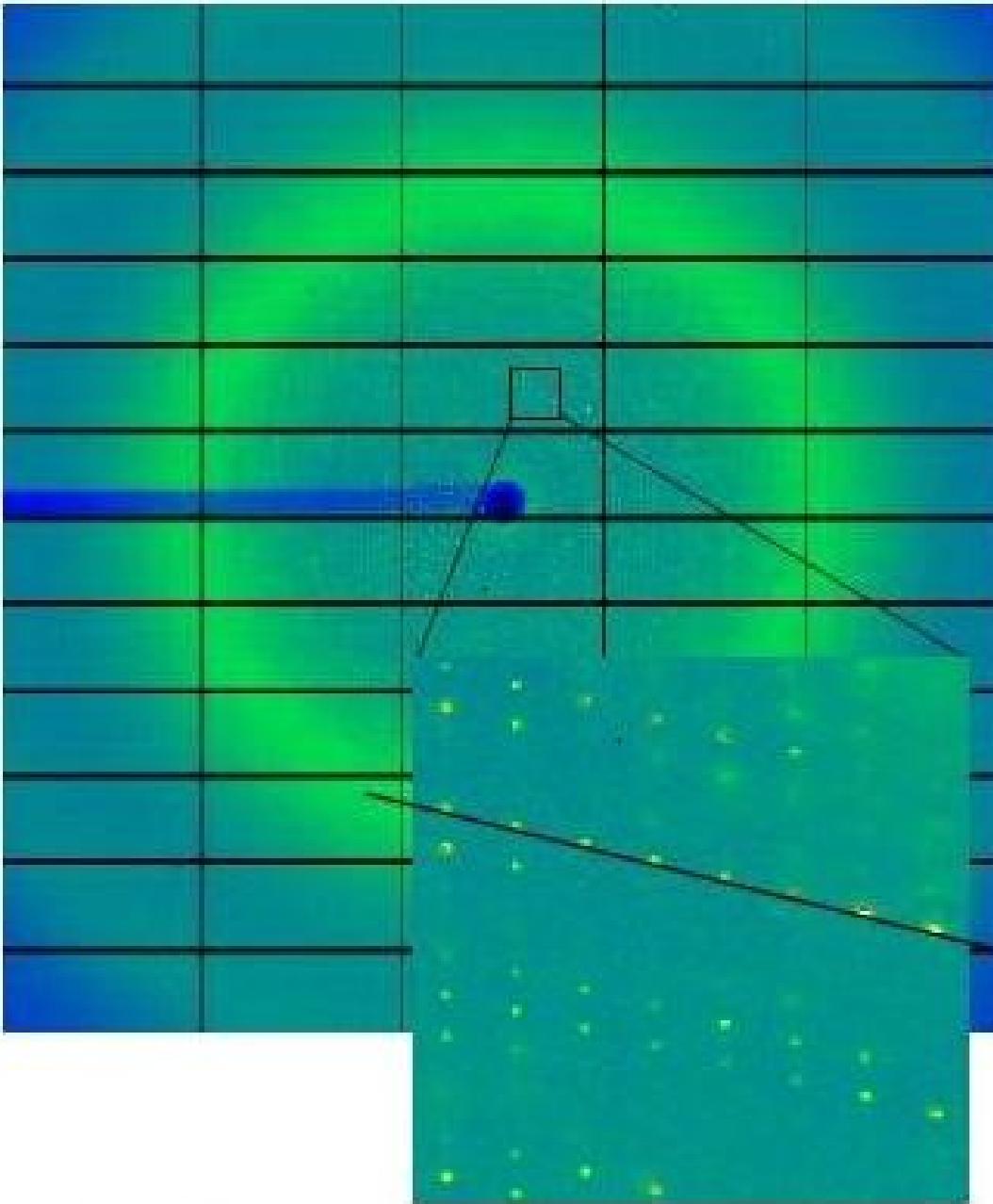


Figure 7: Left: Typical diffraction pattern as produced by a protein crystal. The zoom shows an arbitrary pattern with a large amount of reflections where the intensities vary within several orders of magnitudes (right). ©V. Ramakrishnan, MRC Laboratory of Molecular Biology, Cambridge.

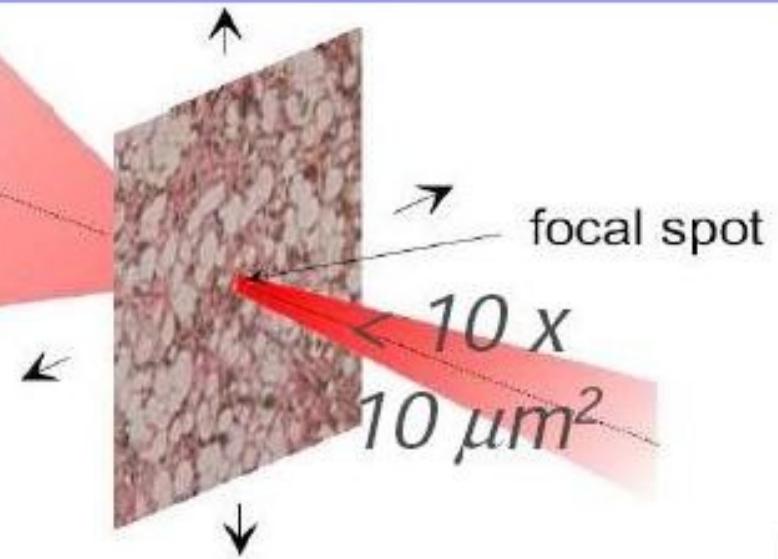


## Scanning SAXS at beamline X012SA of SLS, F.Pfeiffer et. al.

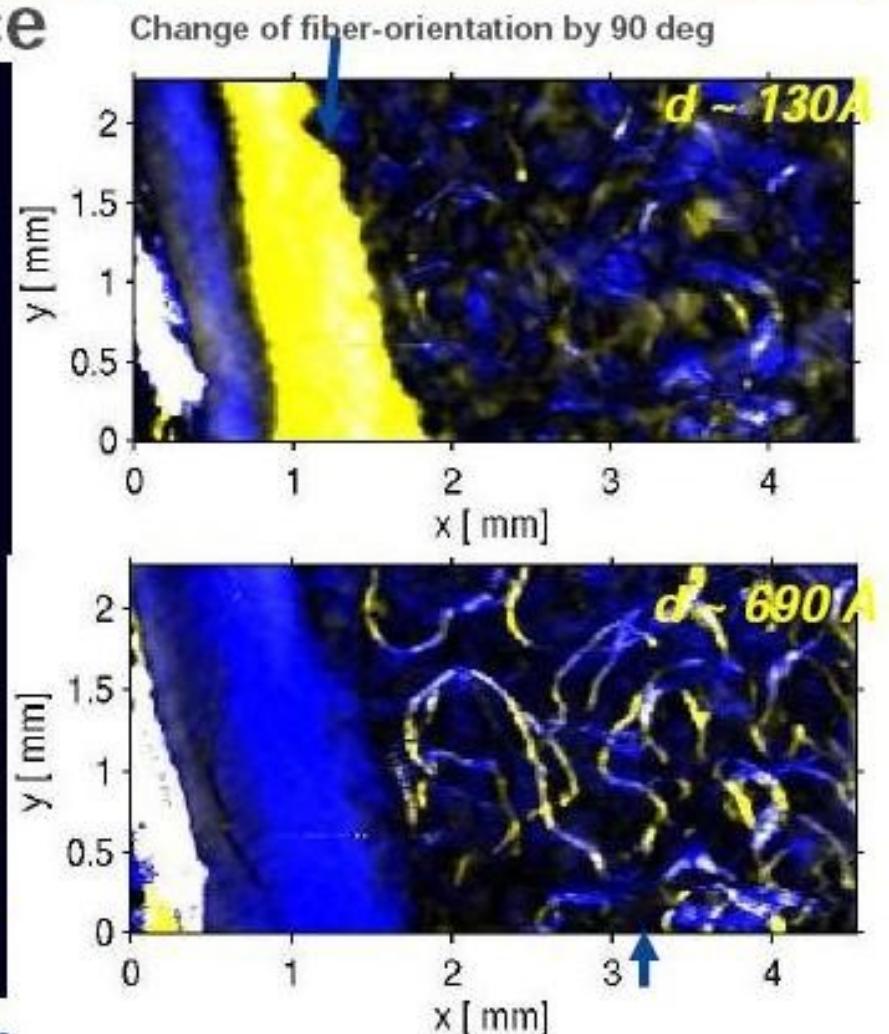
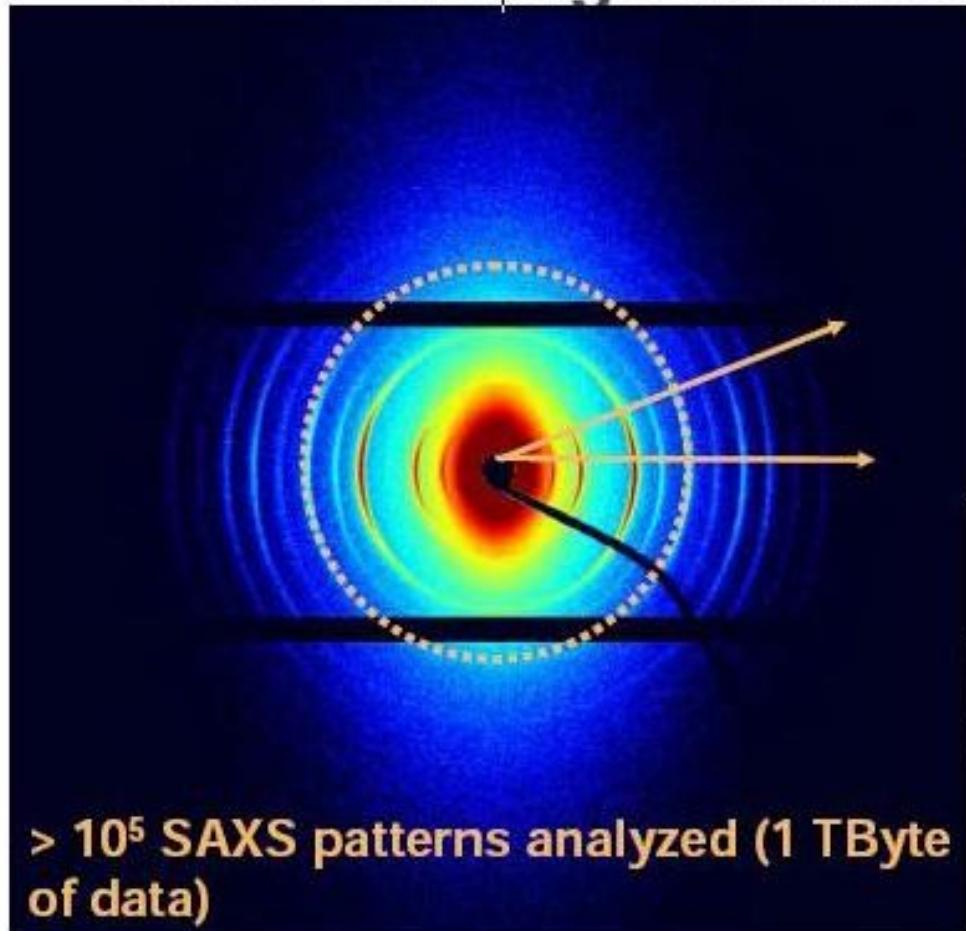
fast framing  
PILATUS 2M



- Automated scans, > 100 x 100 points
- Pilatus 2M used in different acquisition modes:
  - 24 module: full area mode
  - 3x2 module: 2 bank mode
  - 1x3 module: 3 module mode
- Benefit: Higher speed, lower amount of data



## bone cartilage interface



Evaluation of intensity and orientation angle for each pattern & d-spacing >>



**DECTRIS**  
Next Generation X-Ray Detectors

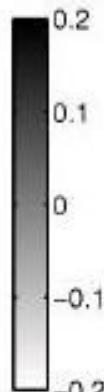


## Comparison: Absorption and Phase Contrast Imaging

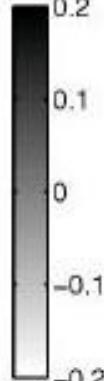
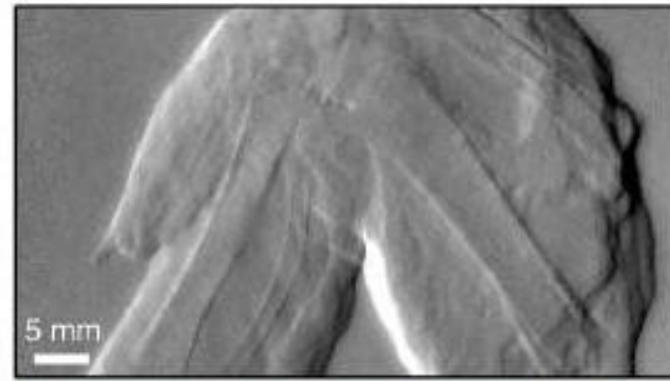
Absorption



Phase-Contrast



CCD



M. Bech, O. Bunk, C. David, P. Kraft, h. Brönnimann, E.F. Eikenberry and F. Pfeiffer,  
X-ray imaging with the PILATUS 100K detector, *Applied Radiation and Isotopes* (2007),  
doi:10.1016/j.apradiso.2007.10.003 [www.dectris.com](http://www.dectris.com)

DECTRIS confidential

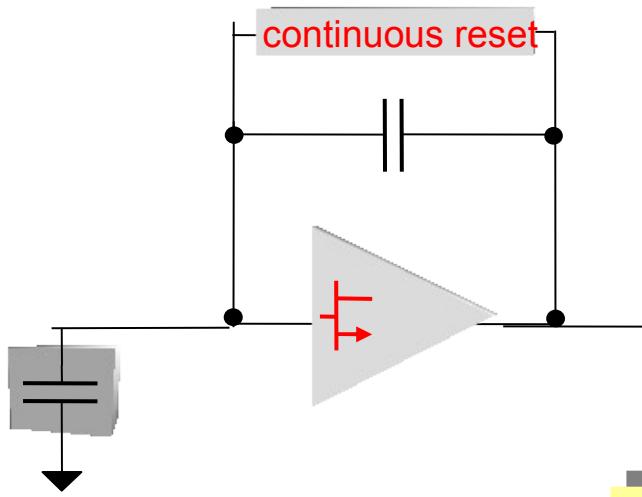
# 1-D detectors

- The complexity of 2-D detectors is not always needed.
  - liquids
  - polycrystalline solids
- Sometimes the openness of a 2-D device causes reduced signal / background
  - UHV environments

# 1-D silicon strip arrays

- 4mm x 0.125mm strips in arrays of 384 and 640 strips
- Fully-depleted 0.4mm thick detectors
- Pitch matched to ASIC, so simple bonding to form arrays
- 350eV energy resolution @ 5.9keV
- 1e5 cps per strip maximum counting rate
- Readout of 640 strips in few ms.
- Two example applications
  - GISAXS
  - Powder diffraction pole figures

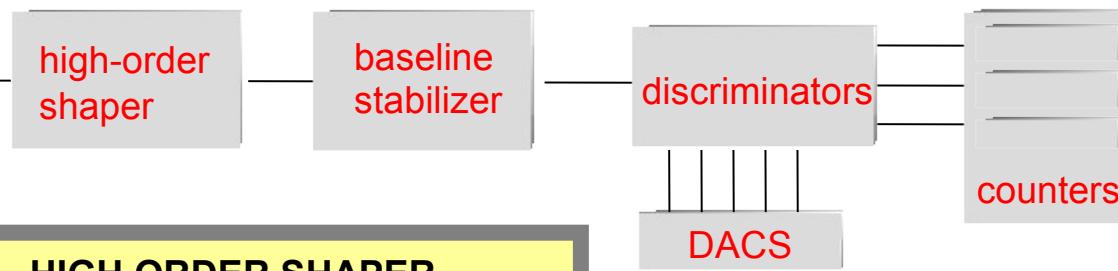
# 'HERMES' ASIC channel overview



**INPUT p-MOSFET**  
 · optimized for operating region  
 · NIM A480, p.713

**CONTINUOUS RESET**  
 · feedback MOSFET  
 · self adaptive 1pA - 100pA  
 · low noise < 3.5e<sup>-</sup> rms @ 1μs  
 · highly linear < 0.2% FS  
 · US patent 5,793,254  
 · NIM A421, p.322  
 · TNS 47, p.1458

≈ 3 mW



**HIGH ORDER SHAPER**  
 · amplifier with passive feedback  
 · 5<sup>th</sup> order complex semigaussian  
 · 2.6x better resolution vs 2<sup>nd</sup> order  
 · TNS 47, p.1857

**BASELINE STABILIZER (BLH)**  
 · low-frequency feedback, BGR  
 · slew-rate limited follower  
 · DC and high-rate stabilization  
 · dispersion < 3mV rms  
 · stability < 2mV rms @ rt×tp<0.1  
 · TNS 47, p.818

≈ 5 mW

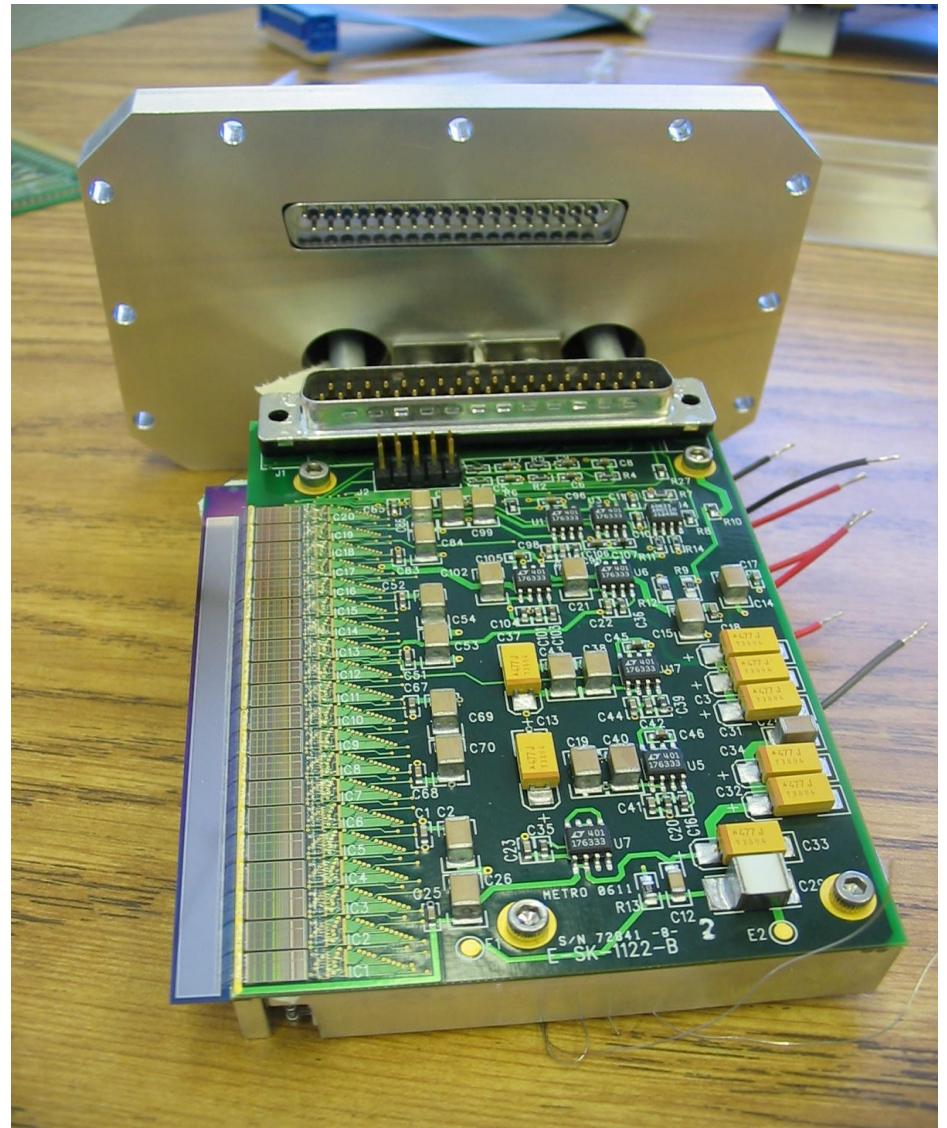
**DISCRIMINATORS**  
 · five comparators  
 · 1 threshold + 2 windows  
 · four 6-bit DACs (1.6mV step)  
 · dispersion (adj) < 2.5e<sup>-</sup> rms

**COUNTERS**  
 · three (one per discriminator)  
 · 24-bit each

ASIC

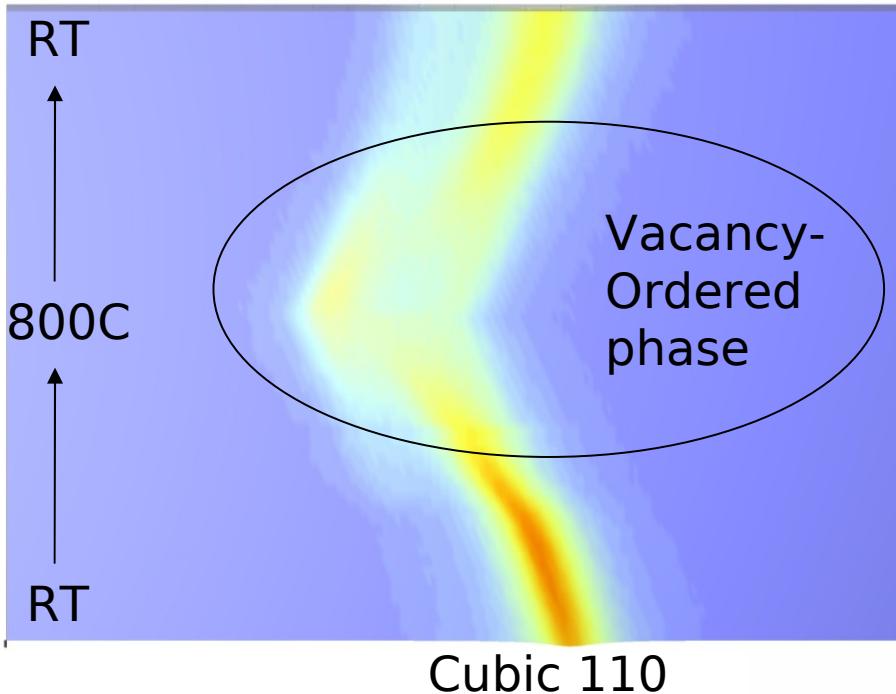
# Microstrip detector

- Diode array (640 strips) at left of picture
- Custom IC's directly to right of strips
- Peltier coolers and water-cooling channels below
- Power regulators and signal buffers to right.
- Diodes cooled to -35C

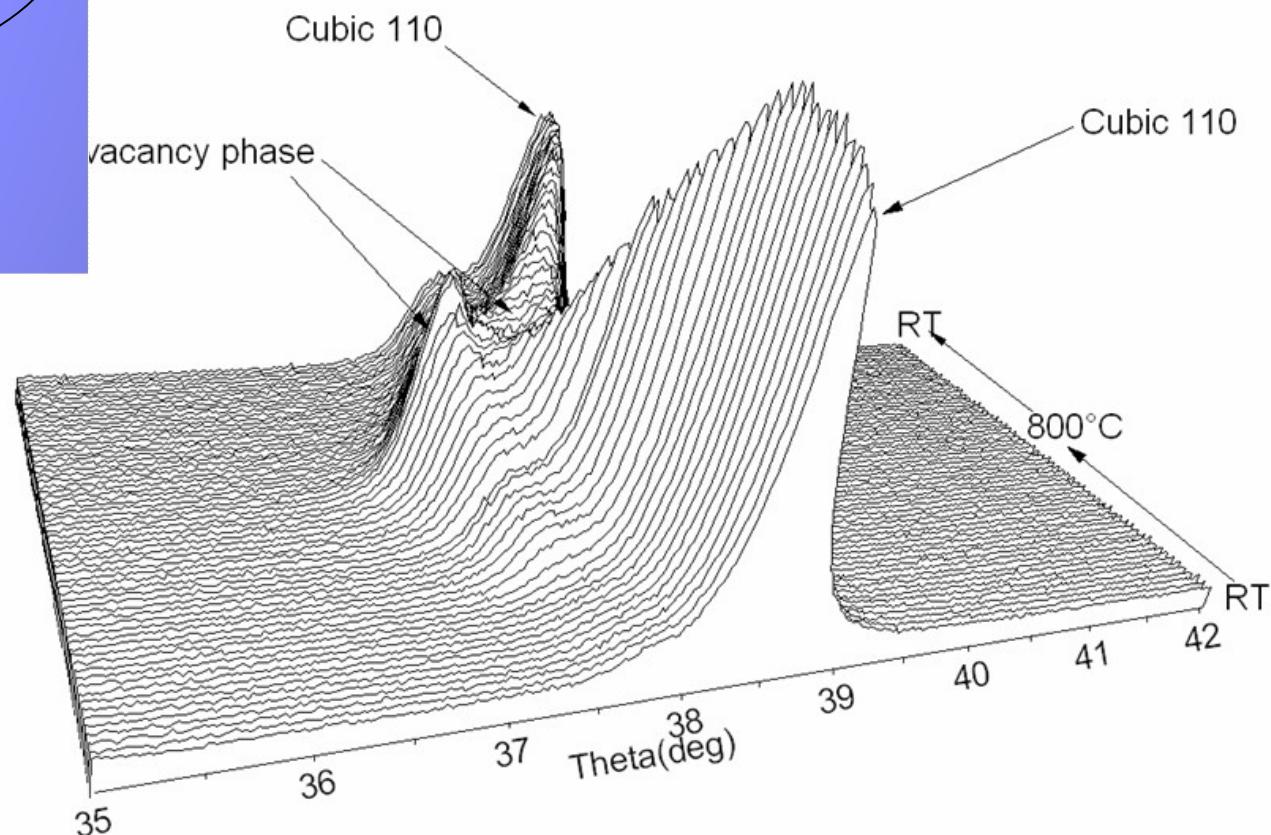


# First direct *in-situ* observation of oxygen vacancy ordering in $(\text{La},\text{Sr})\text{CoO}_{3-\text{d}}$ (and LSCF etc.) cathodes using the Si strip detector

(Alfred University and ORNL)  
Under  $10^{-5}$  atm. oxygen

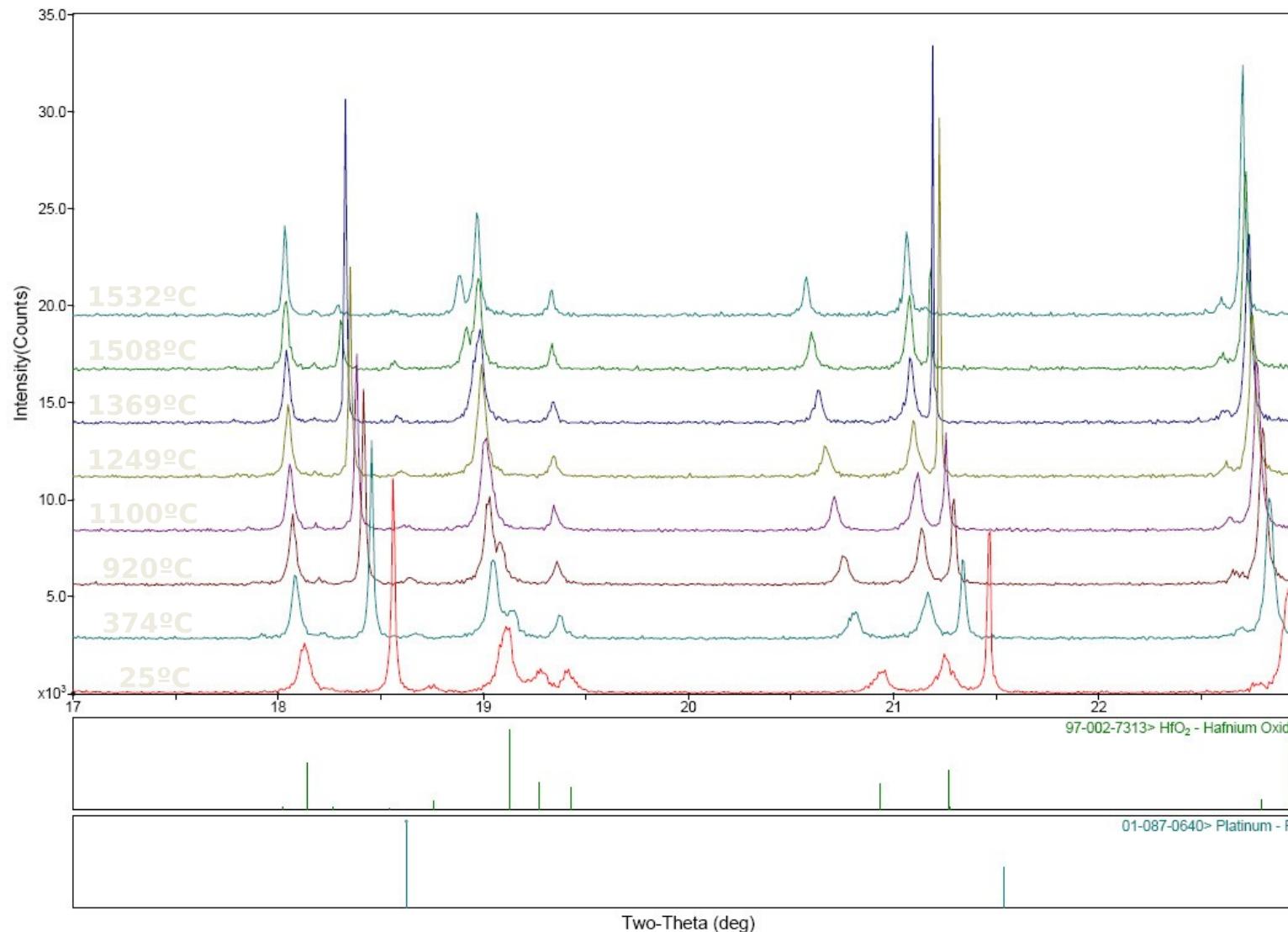


Vacancy ordering **stops**  
ionic conduction



# Thermal Evolution of Hafnia

Department of Materials Science and Engineering  
University of Illinois at Urbana-Champaign



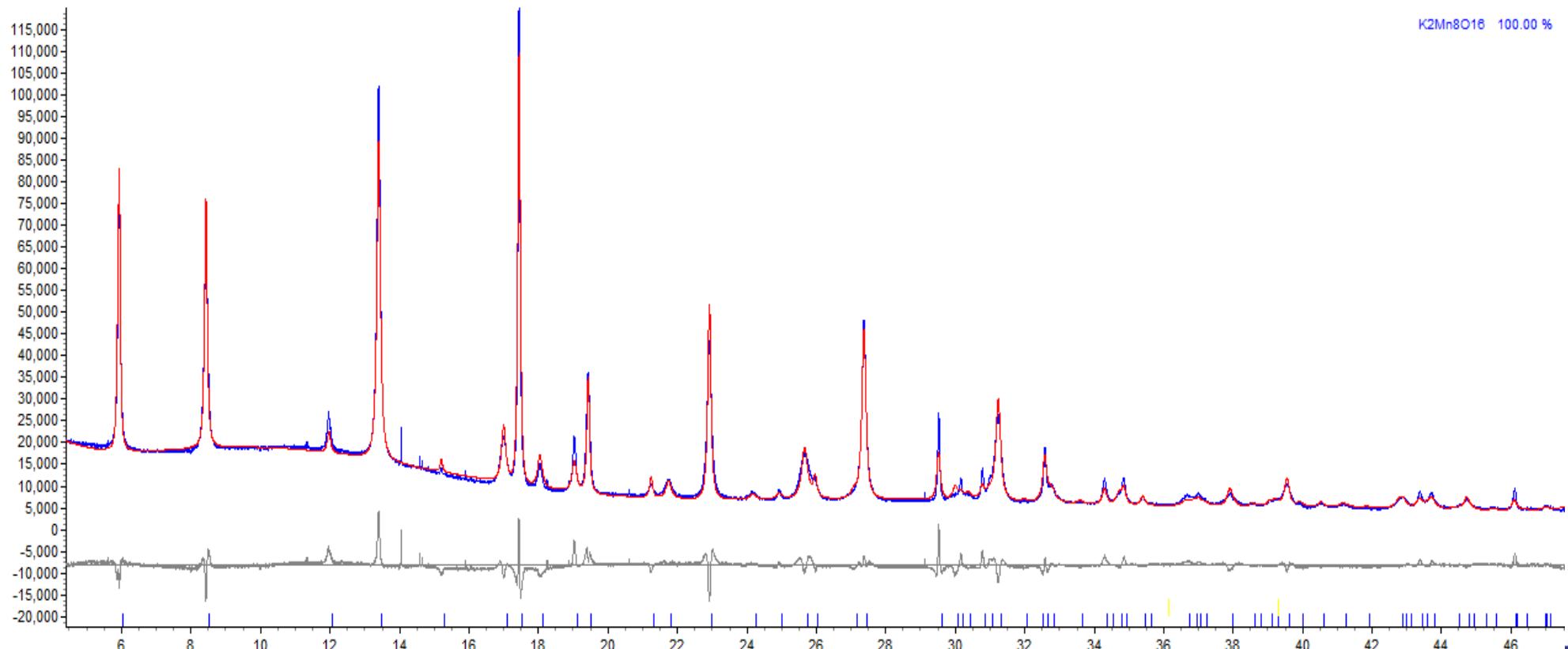
# Structure Refinement Using the Powder XRD Data Taken with The Si Stripe Detector

(University of Connecticut , University of Tennessee and BNL Chemistry)

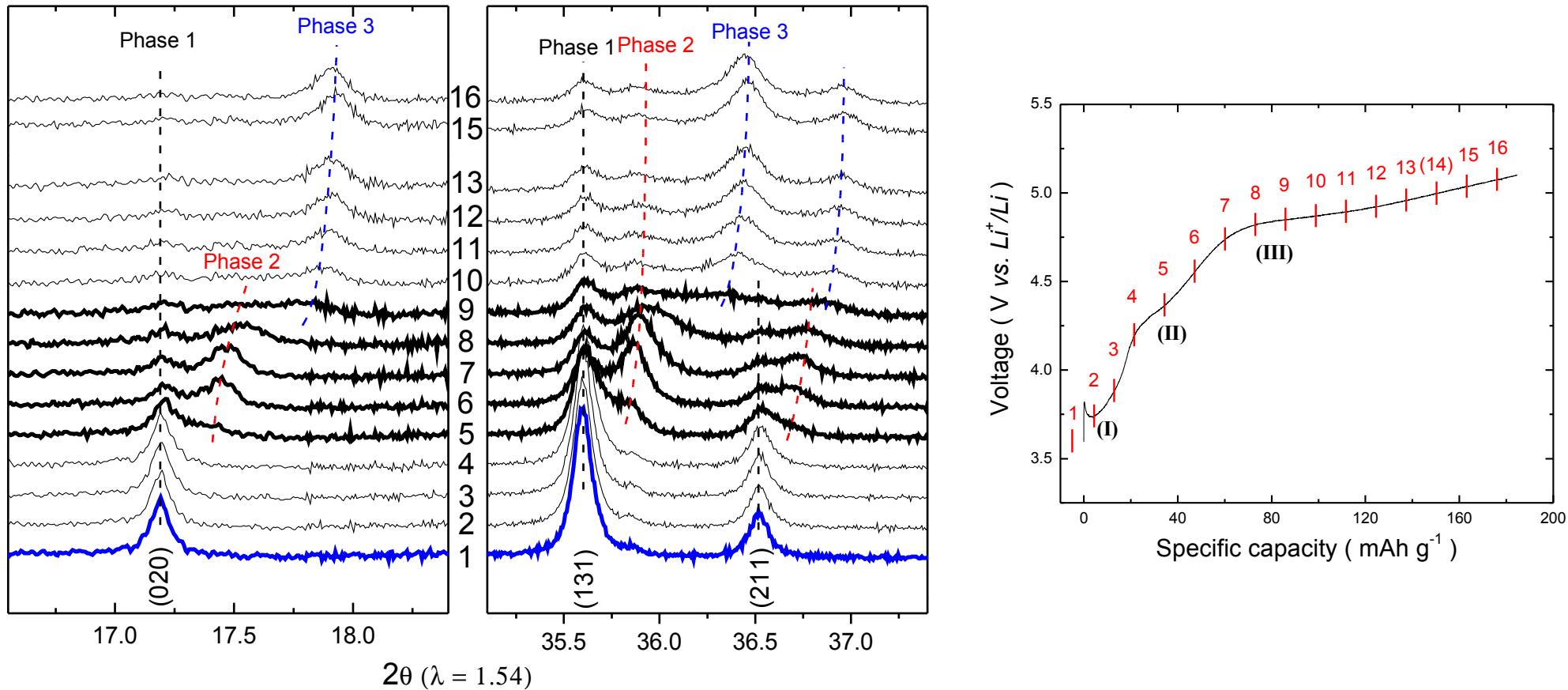
Phase name K<sub>2</sub>Mn<sub>8</sub>O<sub>16</sub> (Cryptomelane)

X-ray wave length 0.73143 Å, Space Group I4/M

a = 9.8480(4), b = 2.8630(1)



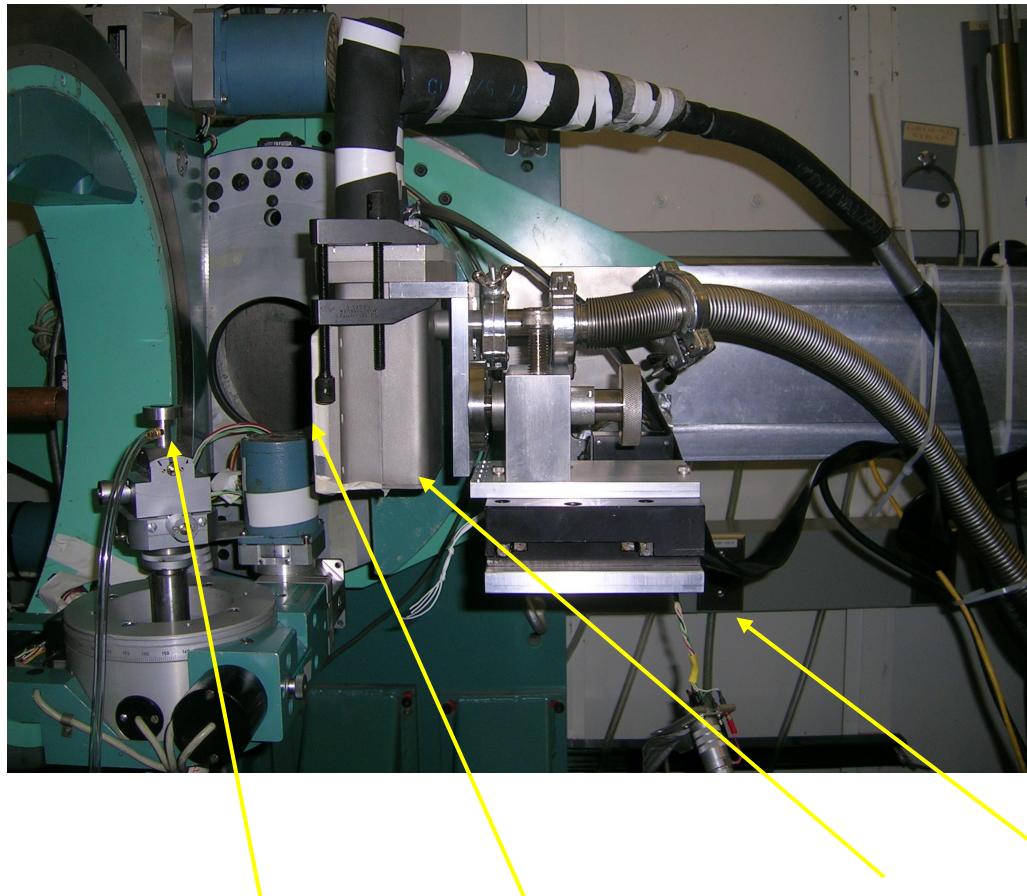
# In situ synchrotron x-ray diffraction studies on LiFe<sub>1/4</sub>Mn<sub>1/4</sub>Co<sub>1/4</sub>Ni<sub>1/4</sub>PO<sub>4</sub> cathodes for Lithium batteries (BNL Chemistry )



(Left) In Situ XRD patterns of C-LiFe<sub>1/4</sub>Mn<sub>1/4</sub>Co<sub>1/4</sub>Ni<sub>1/4</sub>PO<sub>4</sub> during the first charge cycle. Data taken at 17 keV with the  $2\theta$  angle converted to the corresponding values of Cu x-ray tube . The numbers marked beside the patterns correspond to the scan numbers marked on the charge curve (right)

# NSLS beamline X20C (IBM materials research)

C. Detavernier, K. DeKeyser (U. Gent), D.P. Siddons (NSLS), J. Jordan-Sweet, C. Bohnenkamp

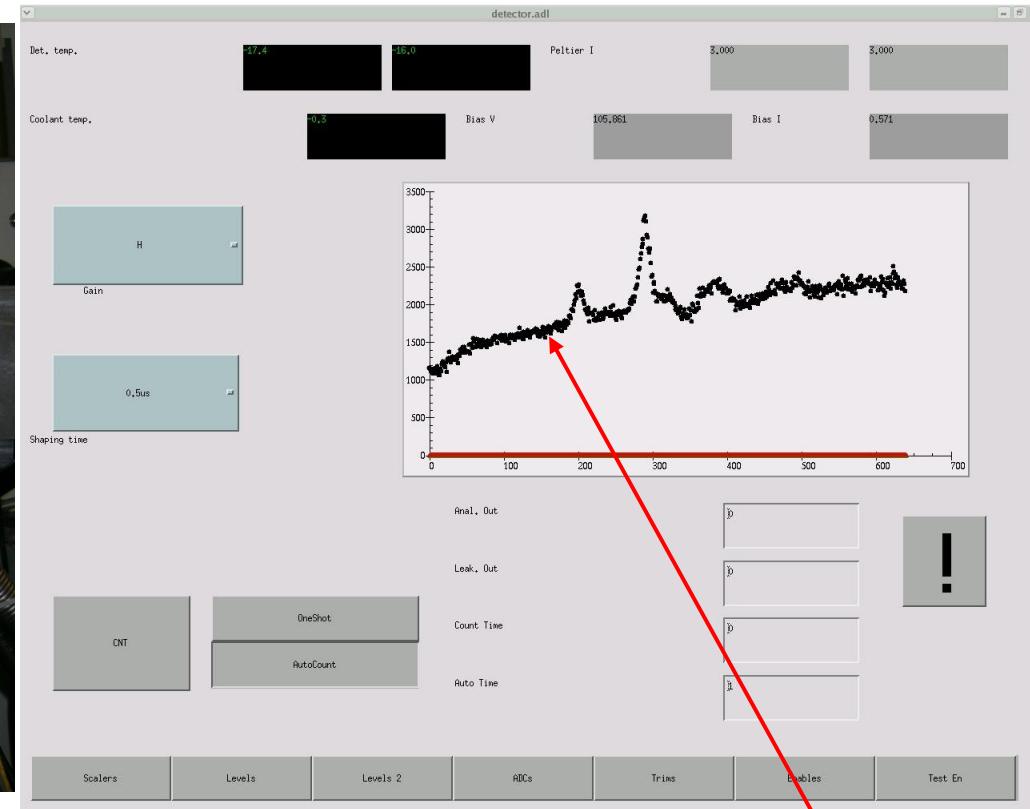


sample stage

detector window

detector chamber

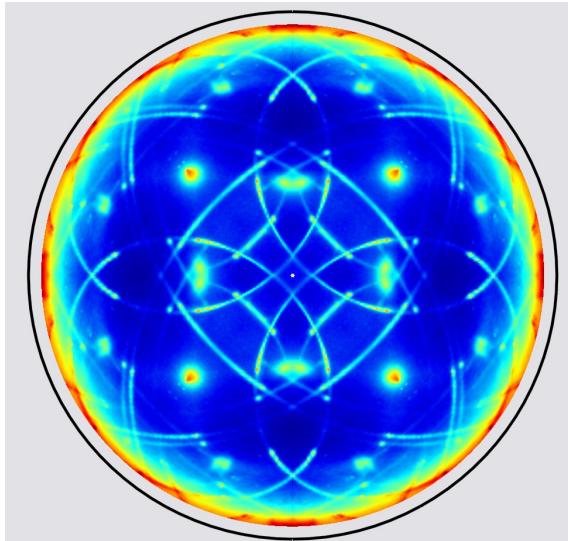
detector mount



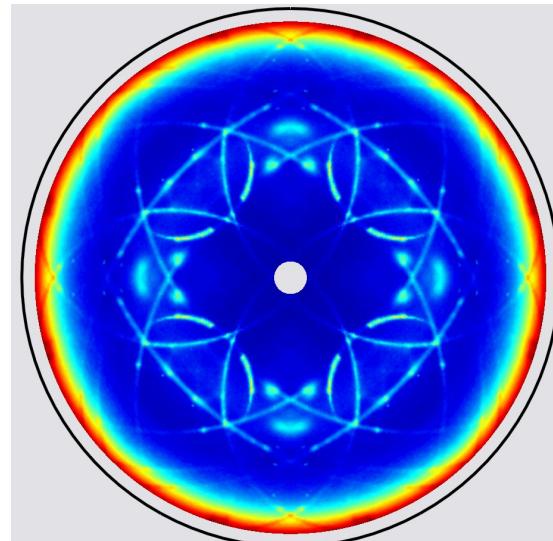
*we now can fit and subtract  
large background*

# First simultaneous pole figures from NSLS linear detector at X20A

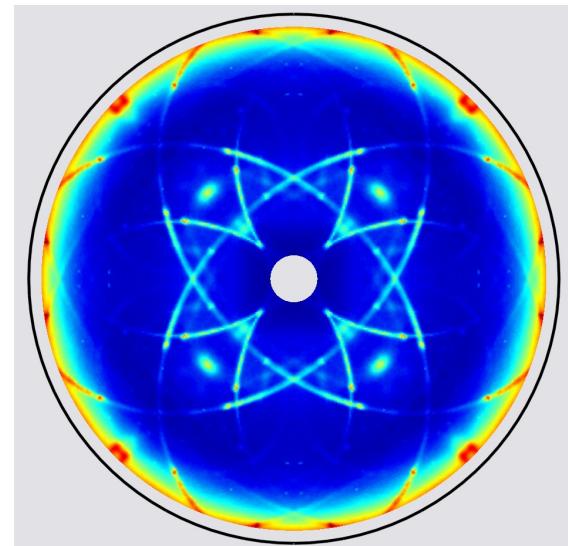
C. Detavernier, K. DeKeyser (U. Gent), D.P. Siddons (NSLS), J. Jordan-Sweet, C. Bohnenkamp



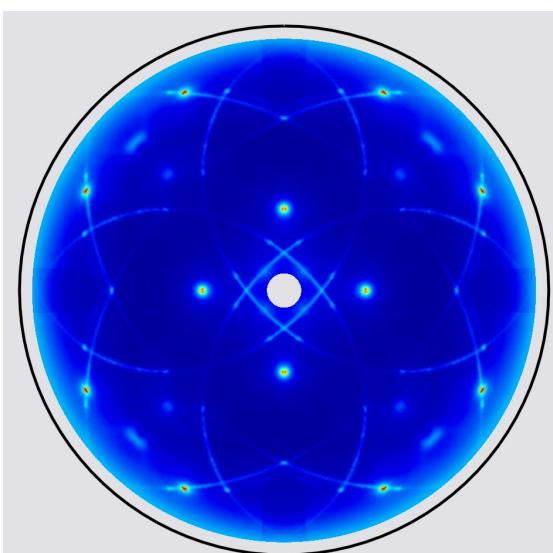
NiSi 112  
 $2\theta = 45.82^\circ$



NiSi 102/111  
 $2\theta = 36^\circ$

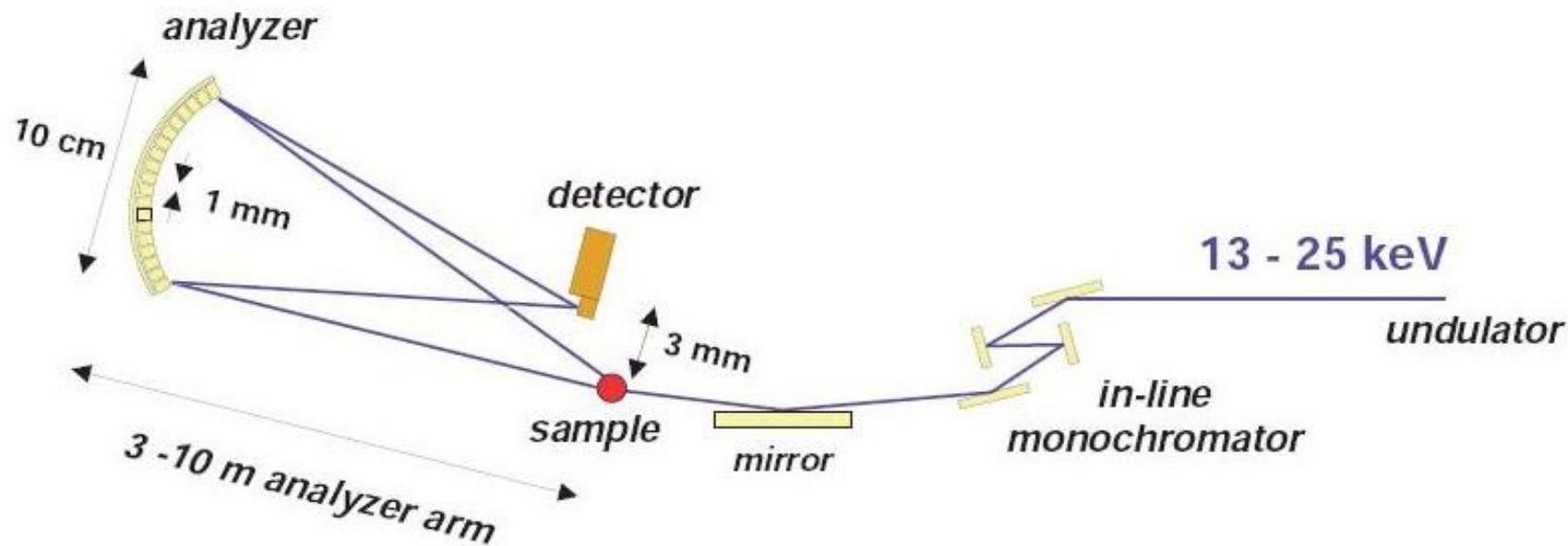


NiSi 002/011  
 $2\theta = 31.5^\circ$  (NiSi/Si(001) tiled from 90° phi segments)



NiSi 013/020  
 $2\theta = 56.4^\circ$

## ***Spectrometers for Inelastic X-ray Scattering***



Energy resolution: 10 - 1 meV

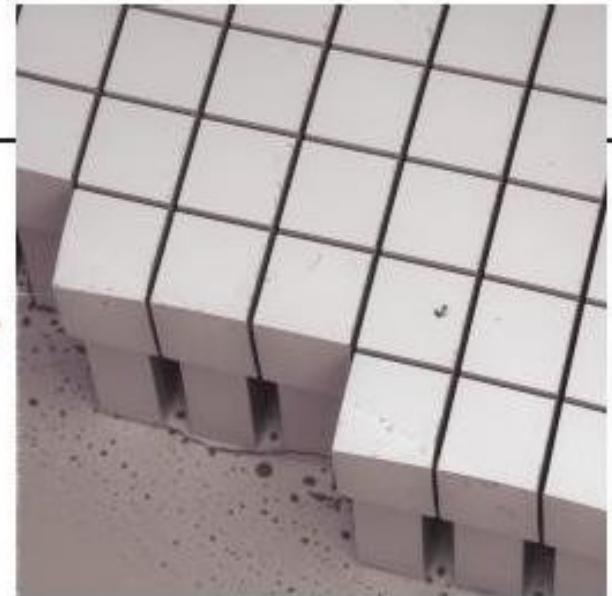
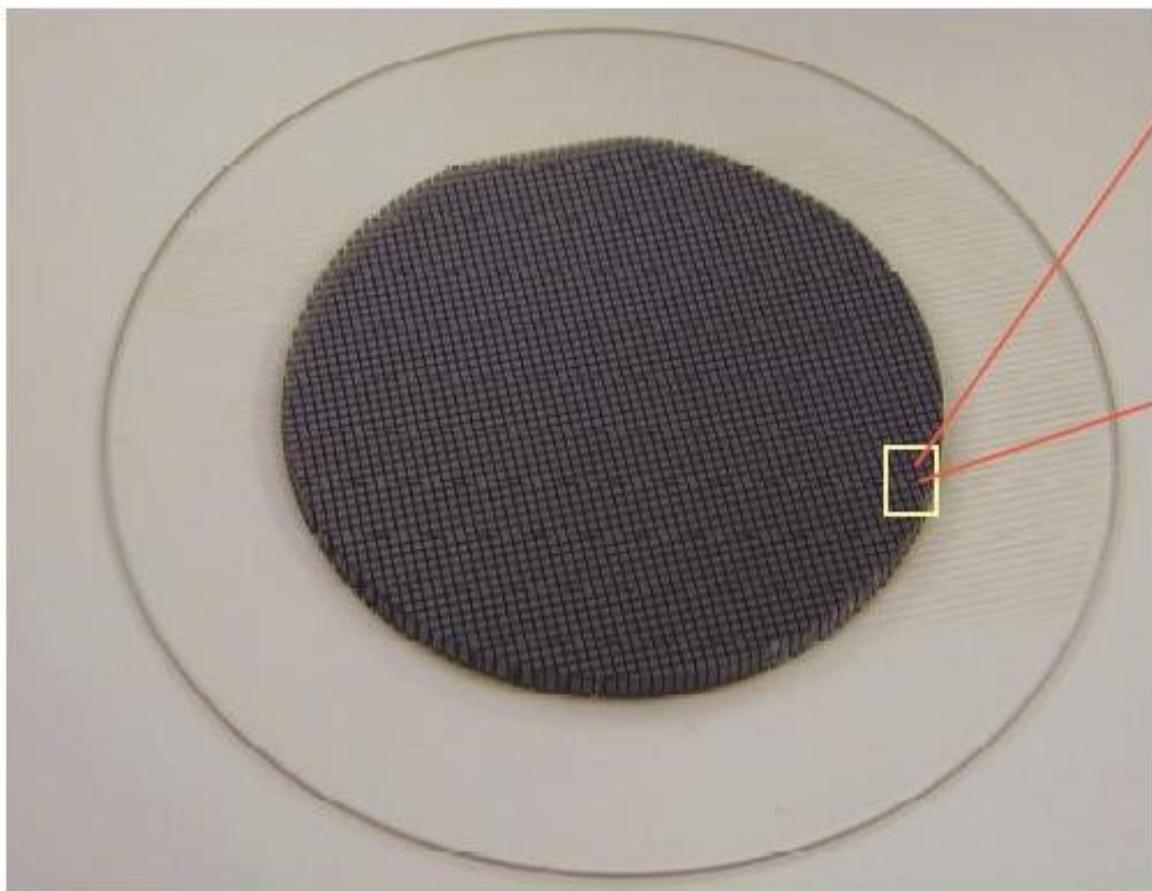
$$\delta E \sim 1/E^4 !$$

*backscattering monochromator  
(ESRF, Sp8)*

2

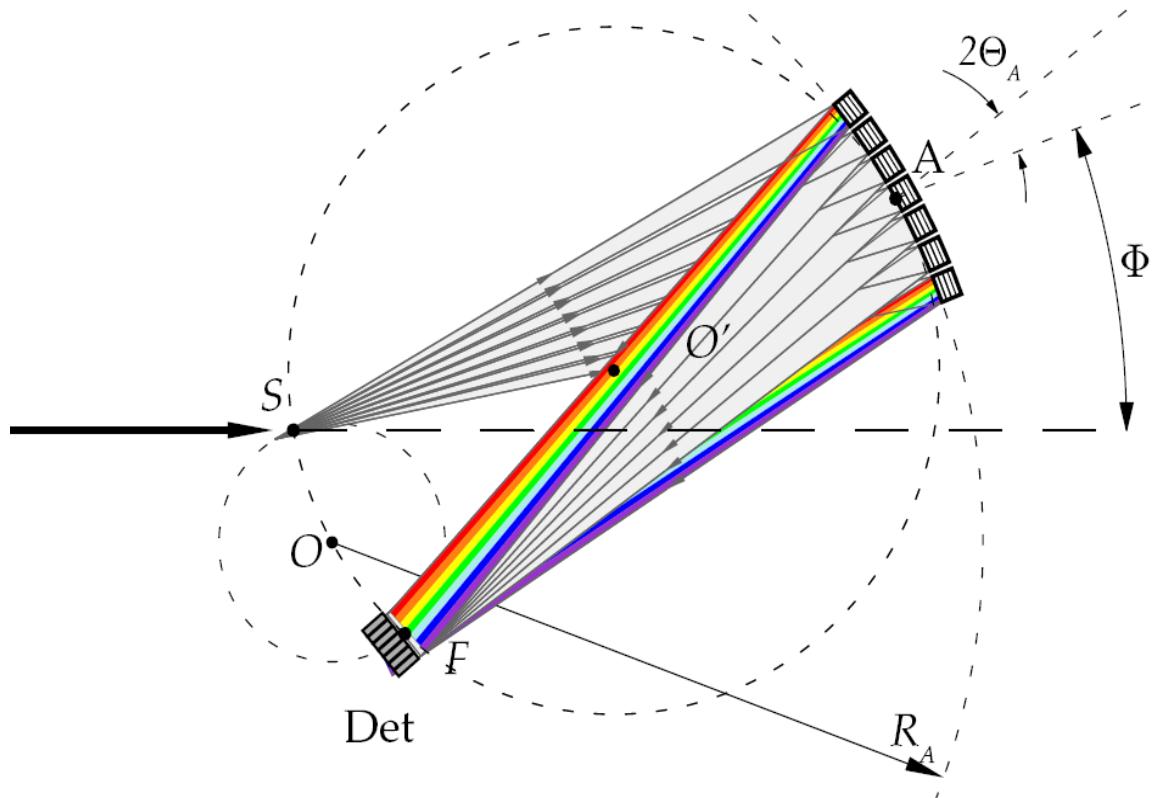
# *Analyzer disk*

Diced and dynamically bent  
silicon substrate



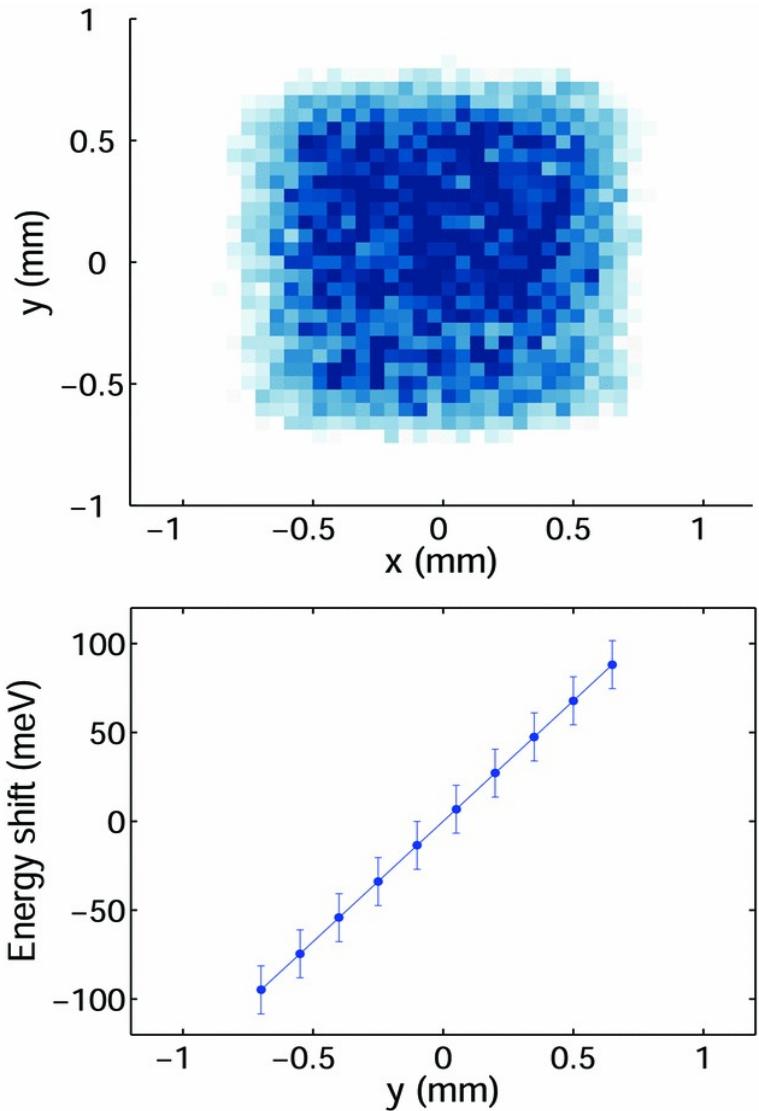
# Inelastic scattering analyzer 'block' dispersion compensation

- Segmented 'spherical' analyzer
- Each 'segment' is mini-Bragg spectrometer
- Can spatially resolve dispersed spectrum from block.



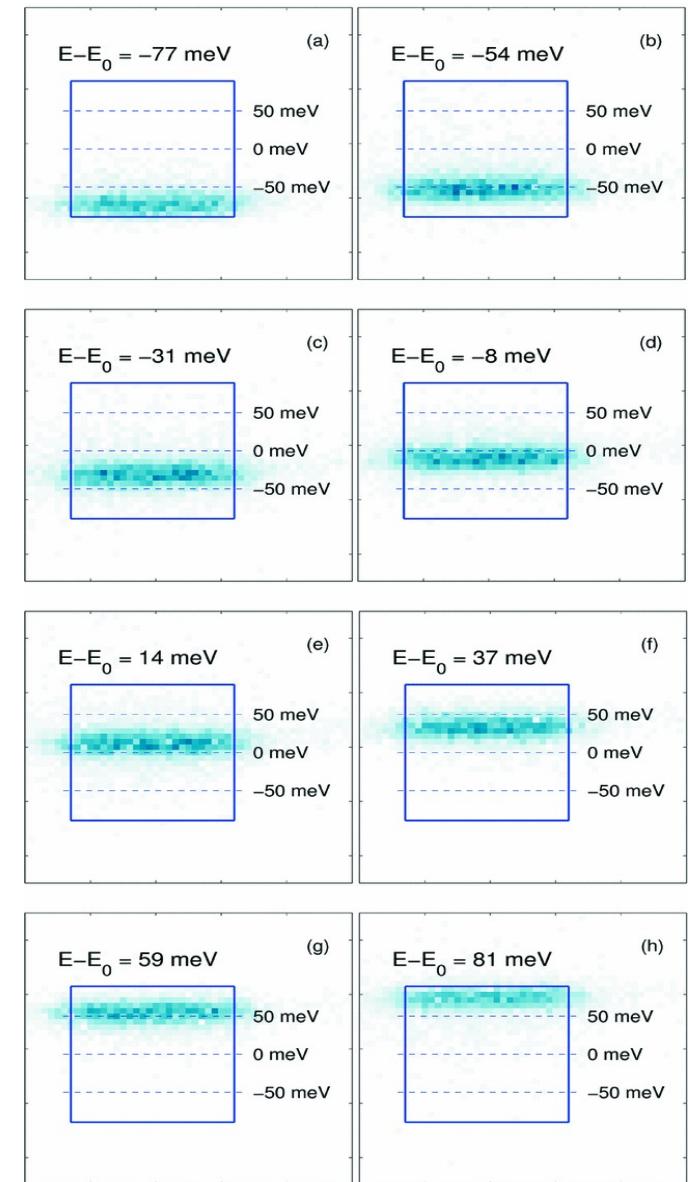
# Dispersion compensation

- S. Huotari et al., J. Synchrotron Rad. (2005). 12, 467-472
- Image of spot at detector
- Single Medipix + silicon sensor
- Shape of spot is x2 image of silicon block.
- Energy correlated with position in vertical dimension



# Direct measurement of dispersion

- Uses high-resolution tuneable monochromator
- Only thing changing is energy of incident beam
- Use of this information provides  $\sim x8$  better resolution
- 1-D detector would work as well in this application

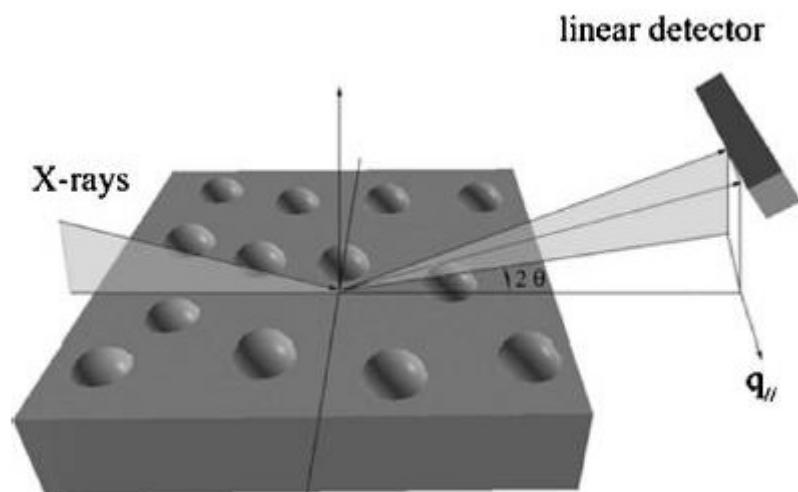


# GISAXS studies of in-situ surface modification

- NSLS beamline X21 has a new in-situ surface chamber.
- Two examples:
  - Ar-ion bombardment of Si surface seeded with Mo nanodots
  - Ga deposition on sapphire

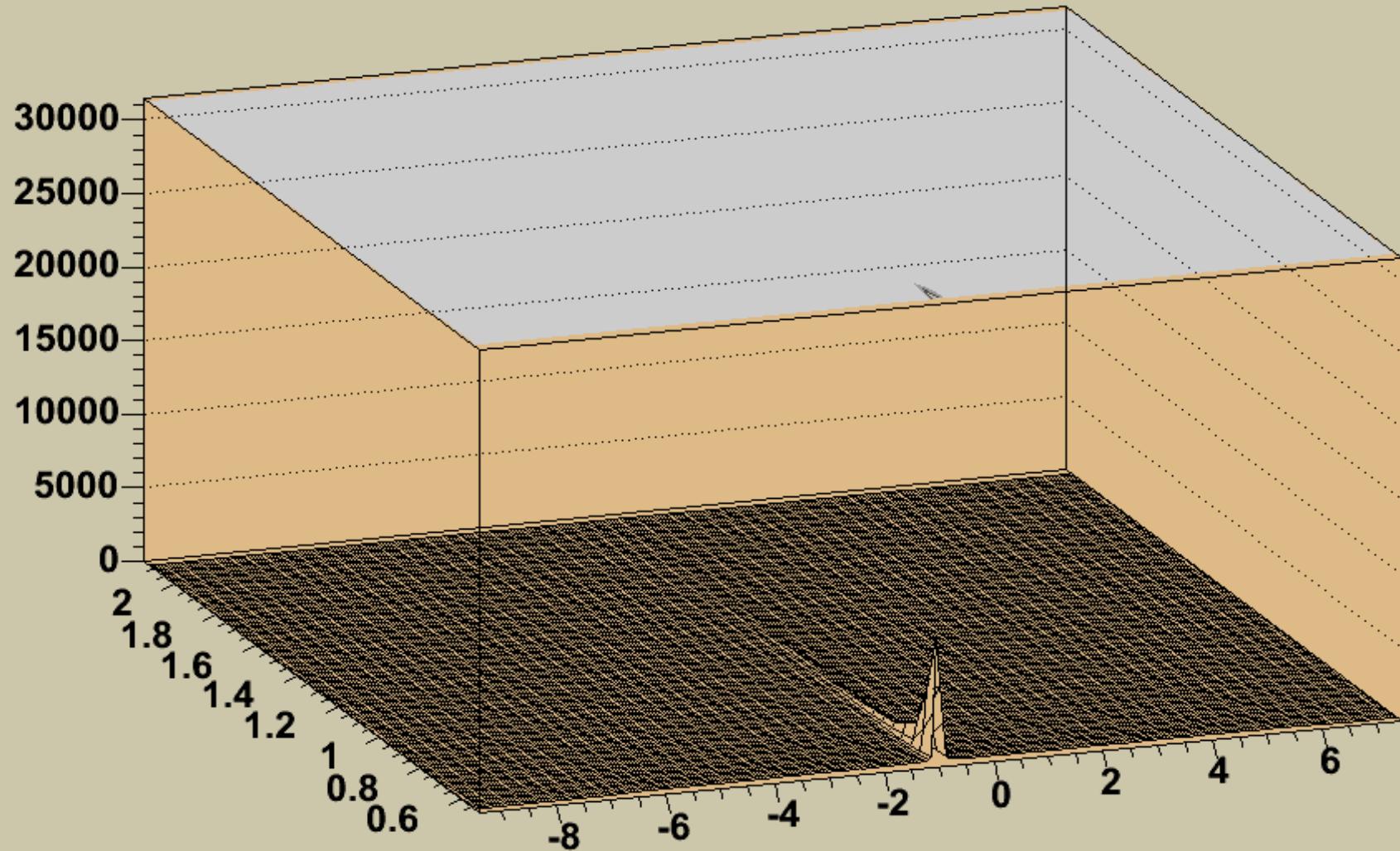
# Grazing-incidence diffraction

- Surface topology on nanometer scale is important: surfaces are different
  - X-rays
- Grazing incidence gives total external reflection
  - No background from substrate
- linear detector set to measure  $q \parallel$
- $q \perp$  by scanning.
- Various surface treatments done under UHV conditions
- 2-D detector has high background
  - “Real-time x-ray studies of gallium adsorption and desorption”. Ahmet S. Ozcan et al., J. Appl. Phys. 100, 084307 (2006)

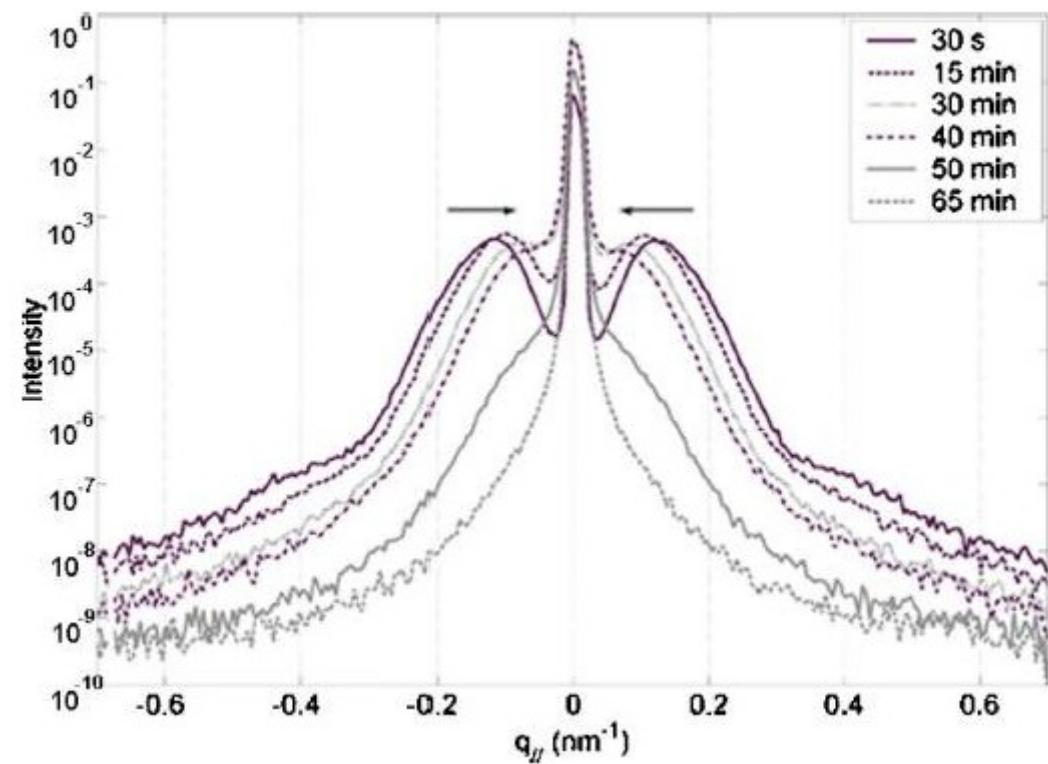
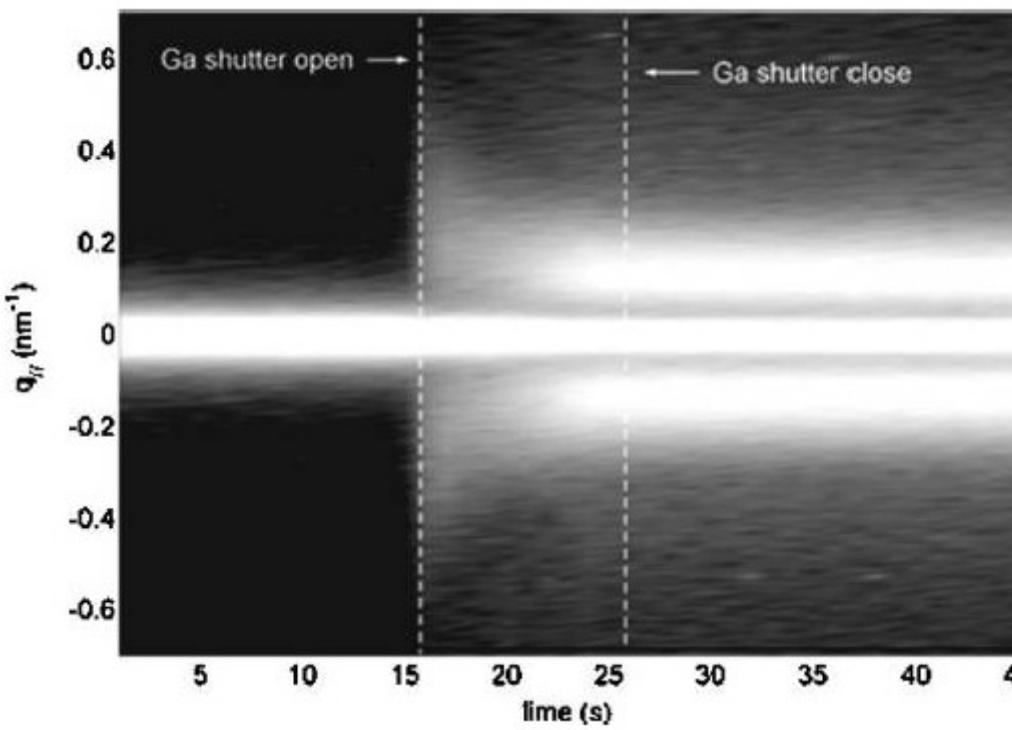


## Roughening of a silicon surface under argon-ion bombardment

In 32 steps



# GIAXS data from Ga droplets on sapphire

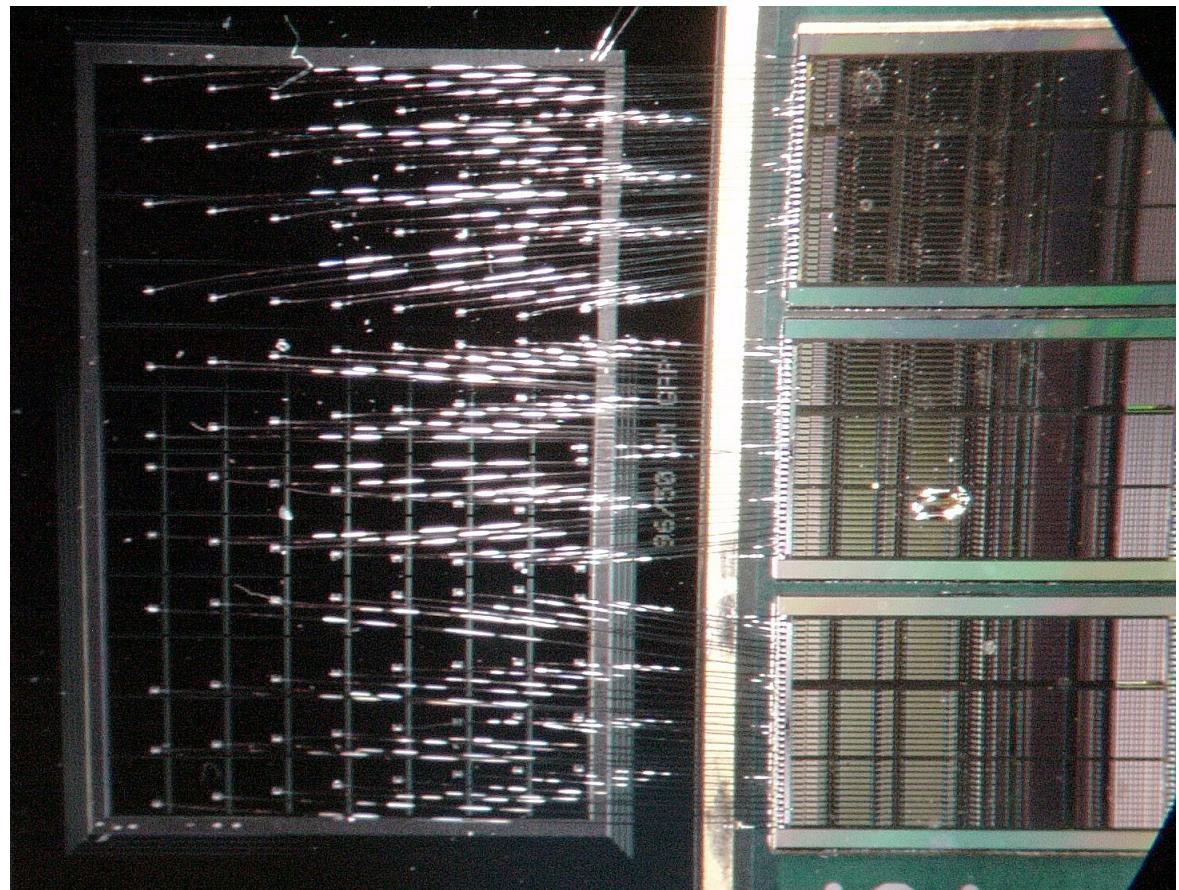


# Pad arrays for spectroscopy

- X-ray fluorescence detection for
  - EXAFS
    - Two hardware pulse-height windows on-chip
    - 24-bit counters on-chip
  - elemental mapping (x-ray microprobe)
    - Full-spectrum acquisition from each of hundreds of detectors
    - Modified ASIC
    - Highly-parallel processing electronics

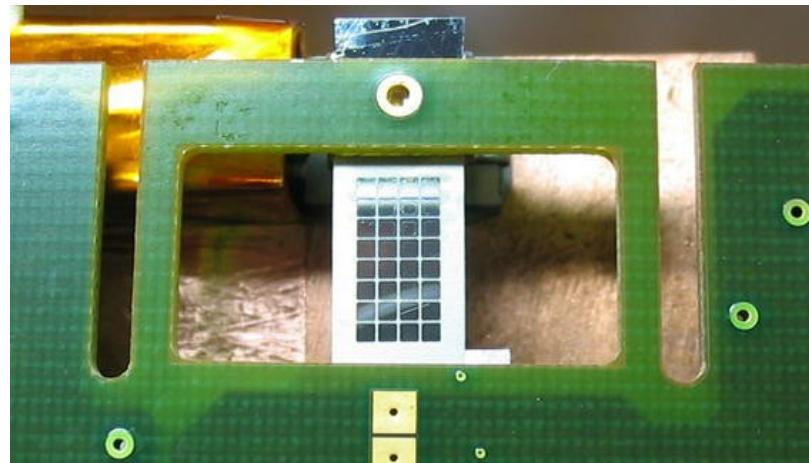
# X-ray Fluorescence Microprobe detector

- 96 pads, 1mm x 1mm, wire-bonded to 3 ASICS.
- The long bonds are rather fragile, but this approach provided least parasitic capacitance.
- Each ASIC provides 32 channels of low-noise analog/digital processing.
- ASIC appears to have 100% yield (no bad channels to date).

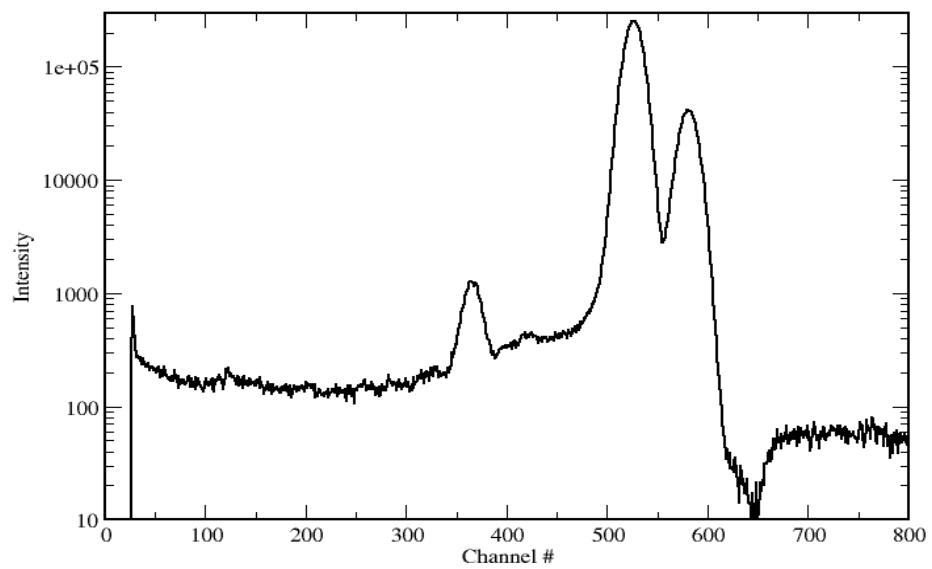


# Avoiding charge-sharing in monolithic pixellated detector

- Charge-sharing near 1mm x 1mm pixel edges significantly degrades peak-valley ratio
- Molybdenum mask shadows inter-pixel region, restoring good p-v ratio.
- Flood  $^{55}\text{Fe}$  spectrum with inter-pixel mask: 1000:1 P/V

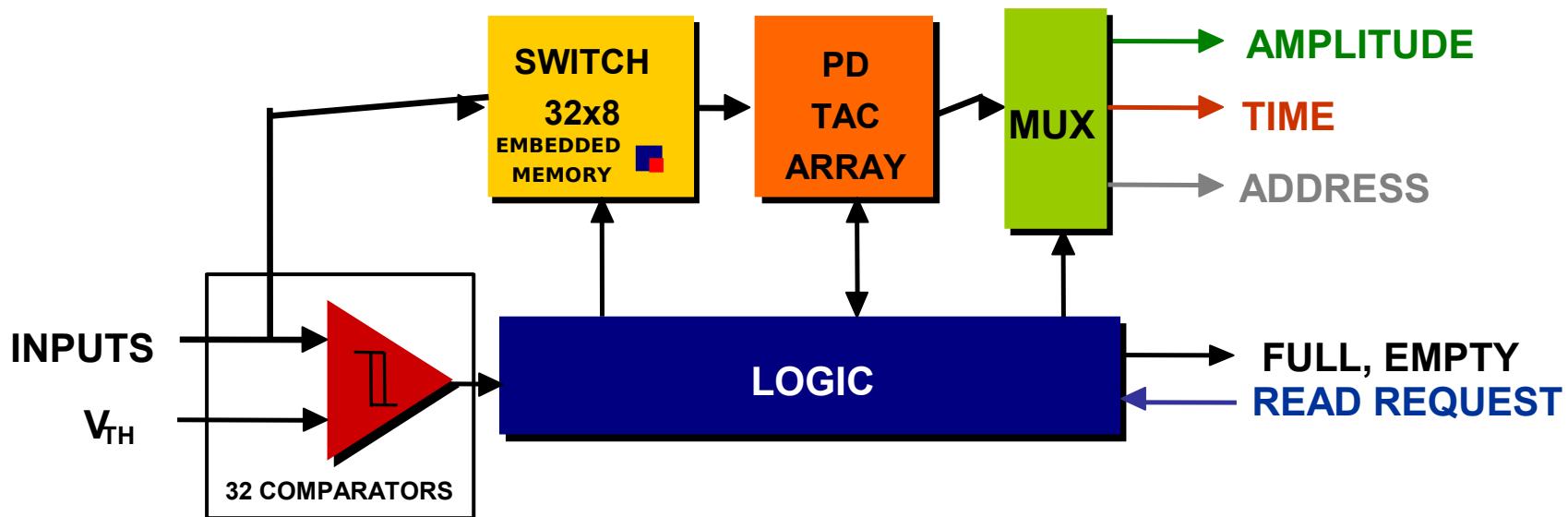


Silicon diode / HERMES Fe55 Spectrum  
with inter-pixel absorber



# SCEPTER: The Peak Detector Derandomizer ASIC

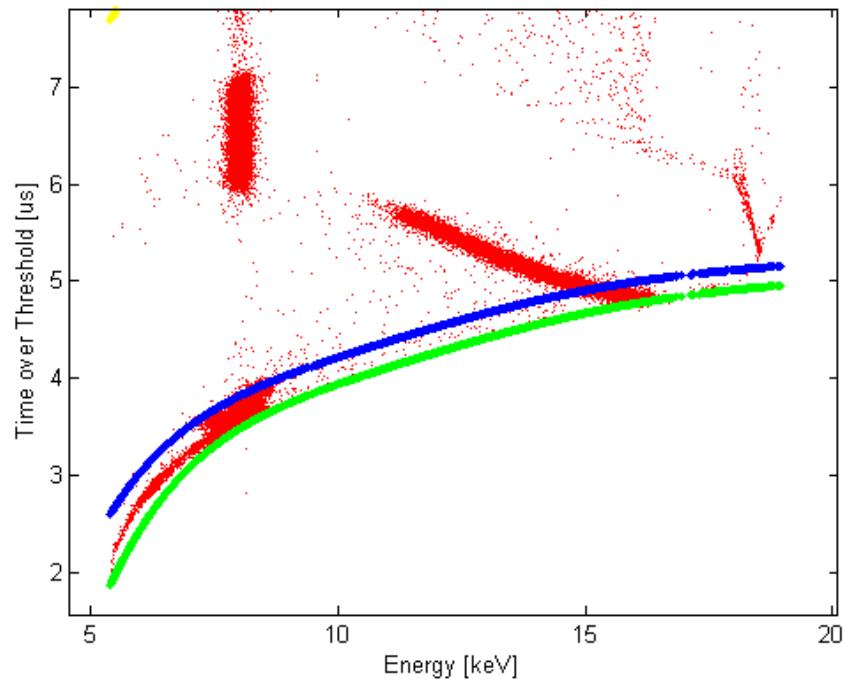
(A. Dragone, G. De Geronimo, P. O'Connor)



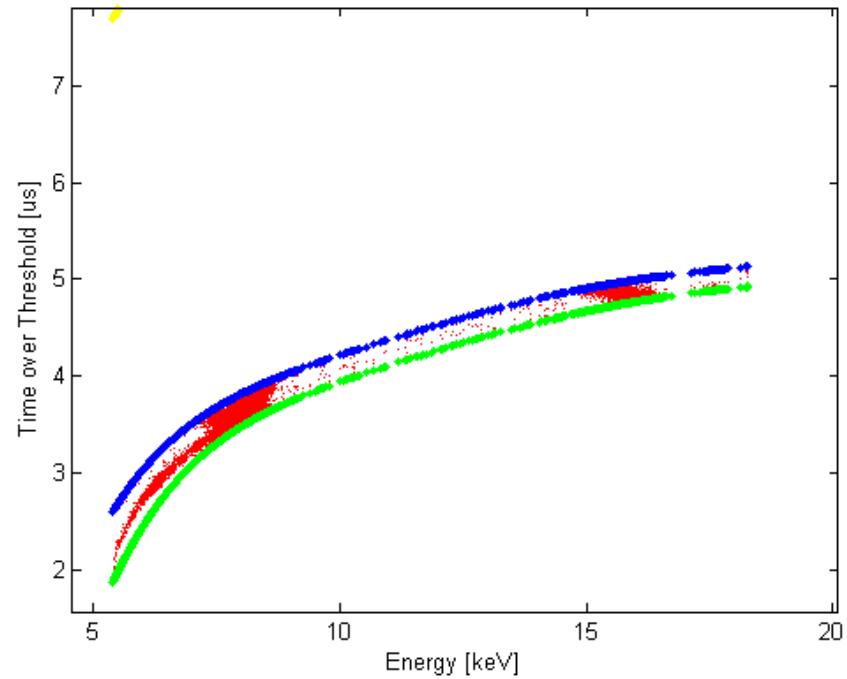
- New architecture for efficient readout of multichannel detectors
  - *Self-triggered and self-sparsifying*
  - *Simultaneous amplitude, time, and address measurement for 32 input channels*
  - *Set of 8 peak detectors act as derandomizing analog memory*
  - *Rate capability improvement over present architectures*
- Based on new 2-phase peak detector combined with Quad-mode TAC
  - *High absolute accuracy (0.2%) and linearity (0.05%), timing accuracy (5 ns)*
  - *Accepts pulses down to 30 ns peaking time, 1.6 MHz rate per channel*
  - *Low power (2 mW per channel)*

# Time-Over-Threshold Measurement for pile-up rejection

## The Pile-Up Rejection Algorithm



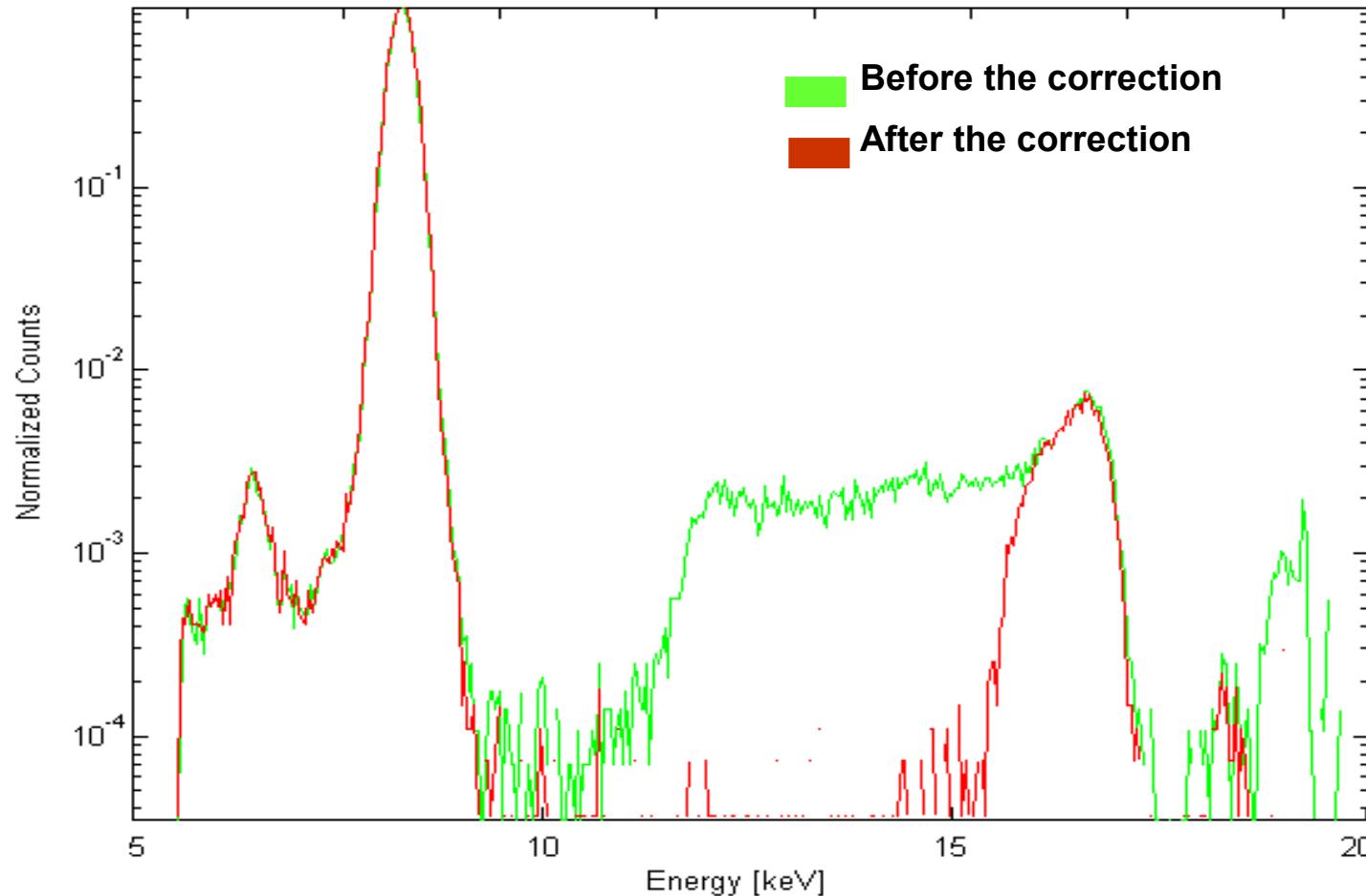
Before The Correction



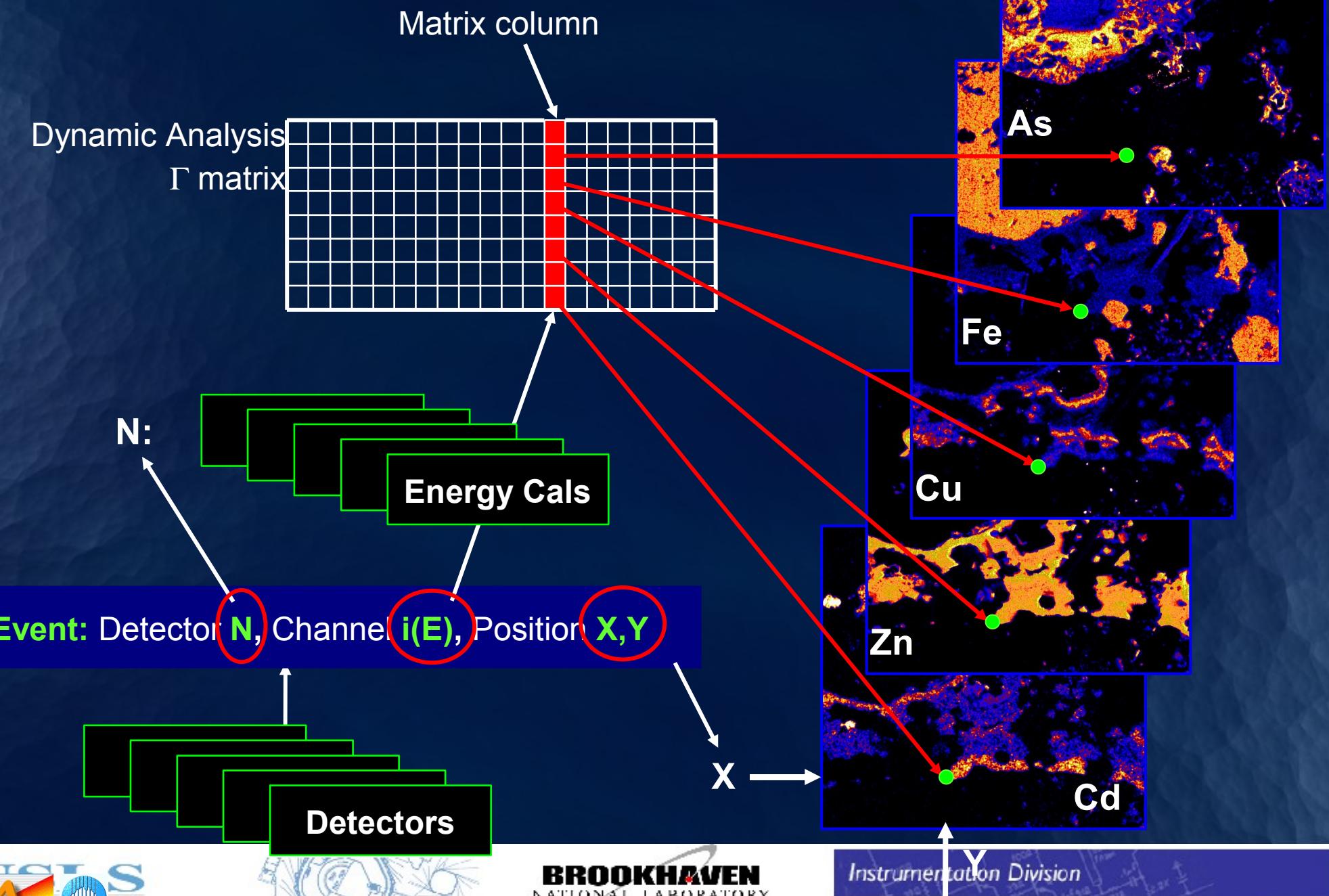
After The Correction

# Time-Over-Threshold Measurement for pile-up rejection

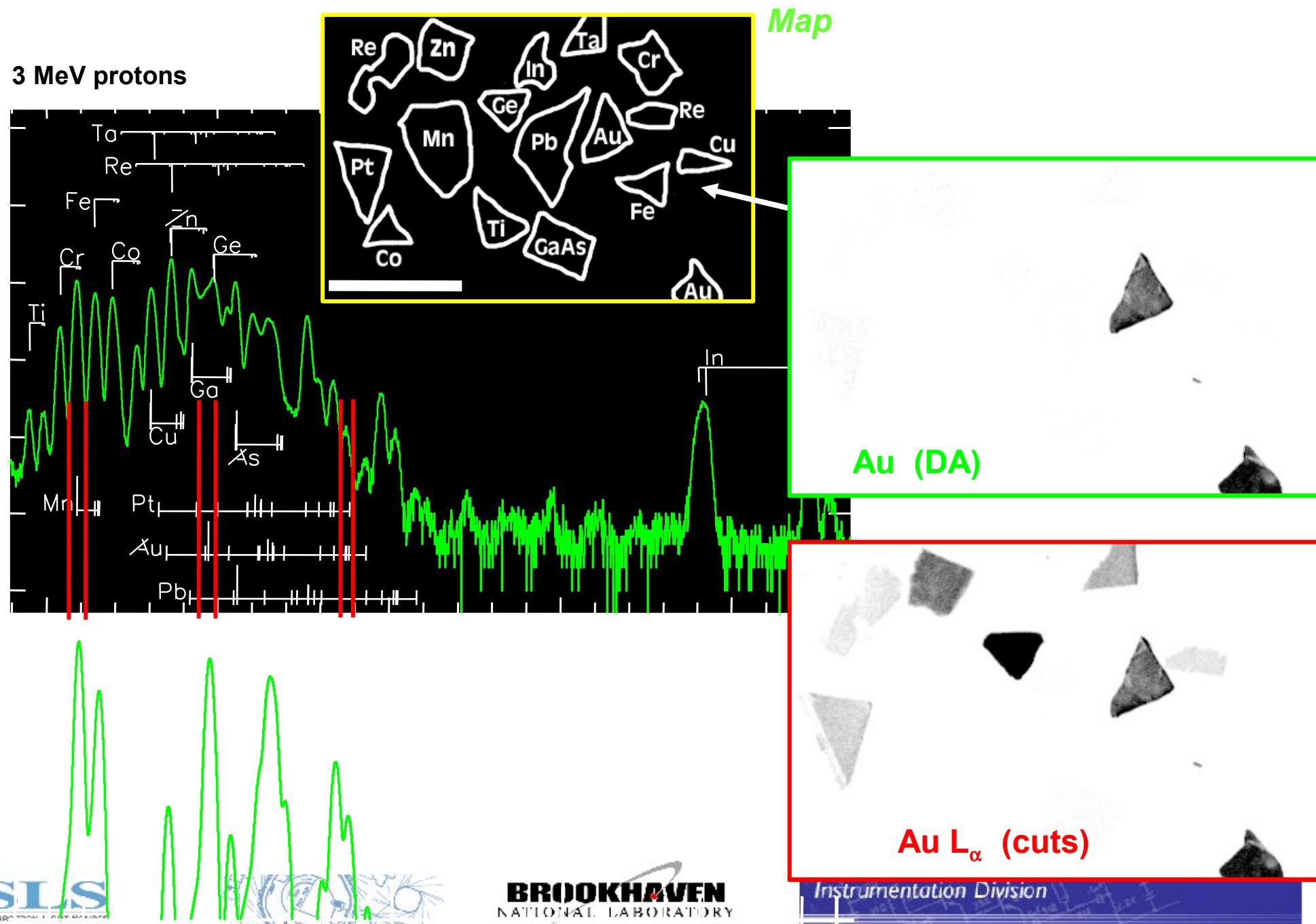
Pulse Height Spectra Comparison



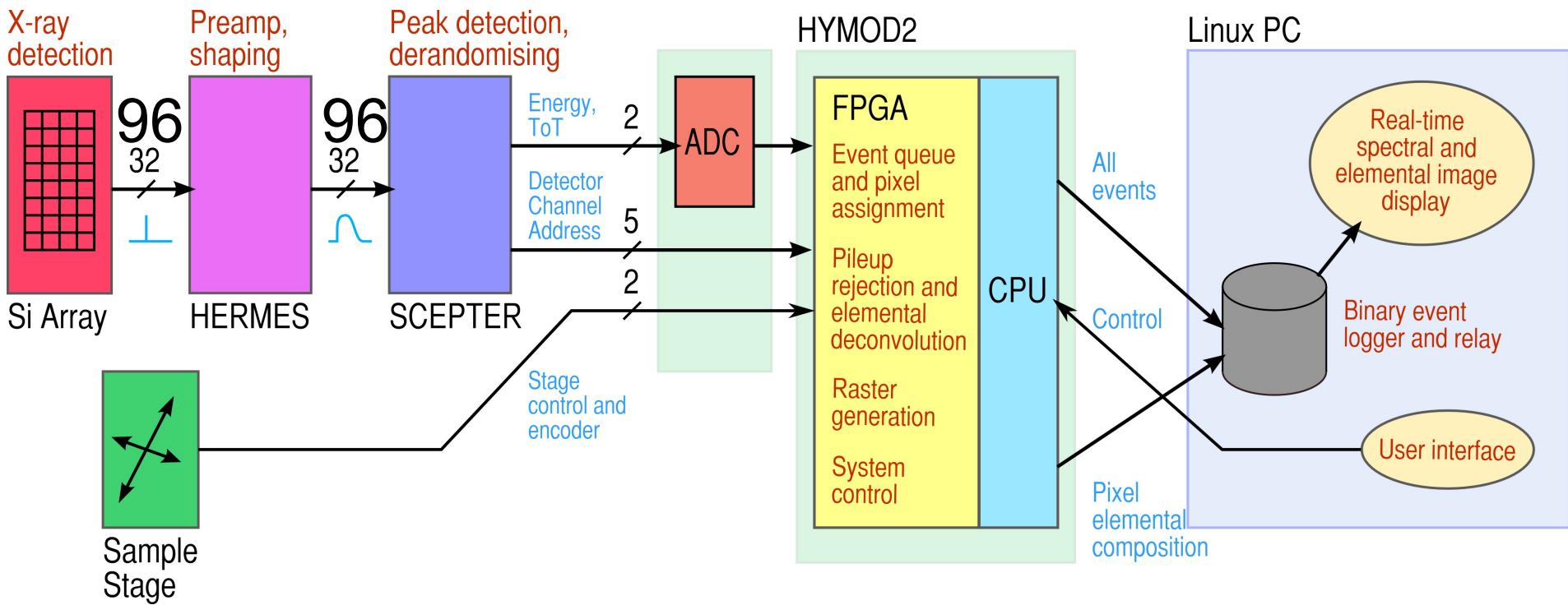
# Real-time Elemental Imaging ...



# Illustration of Dynamic Analysis using PIXE



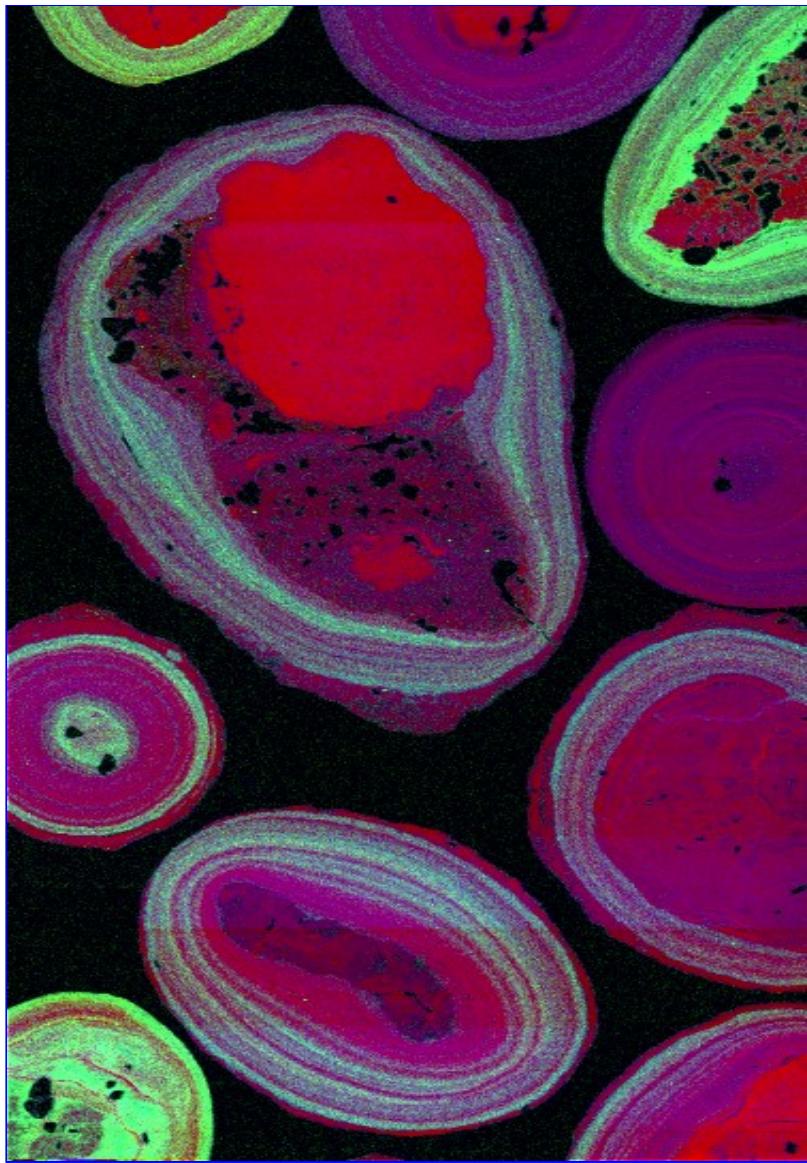
# Demonstration experiment at X27A: Block diagram of test setup



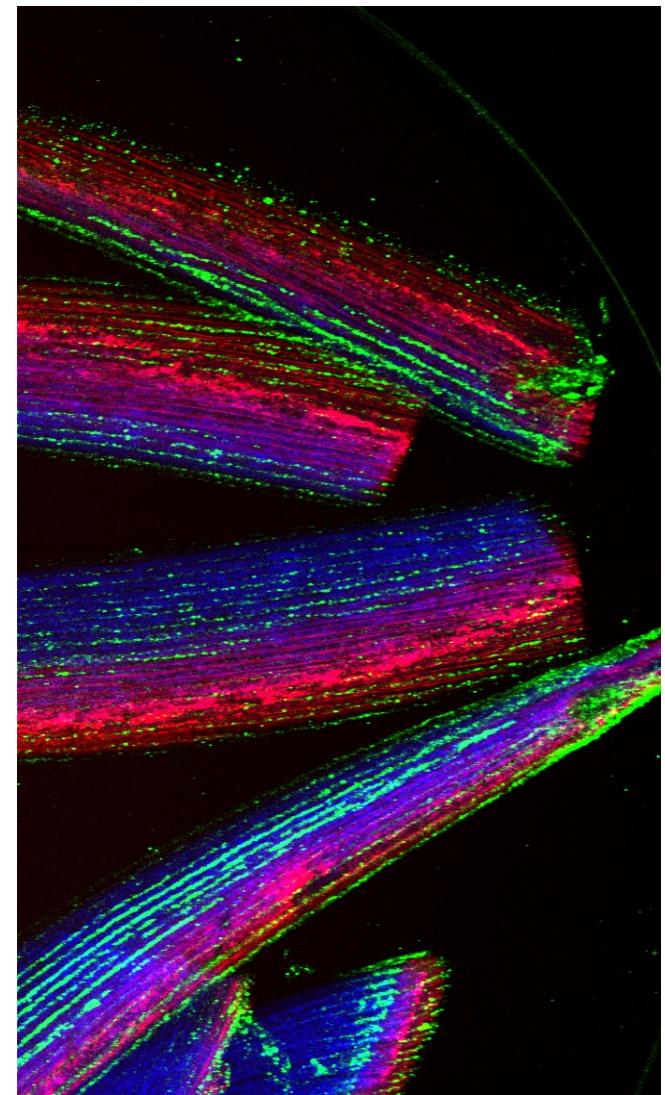
- HYMOD controls stage and reads detector
- Each photon tagged with energy, XY position and pileup status
- Initial coarse scan generates 'average' spectrum which makes DA matrix
- DA technique then presents elemental map as acquisition proceeds.

# Rapid XRF Elemental Mapping (BNL/CSIRO collaboration)

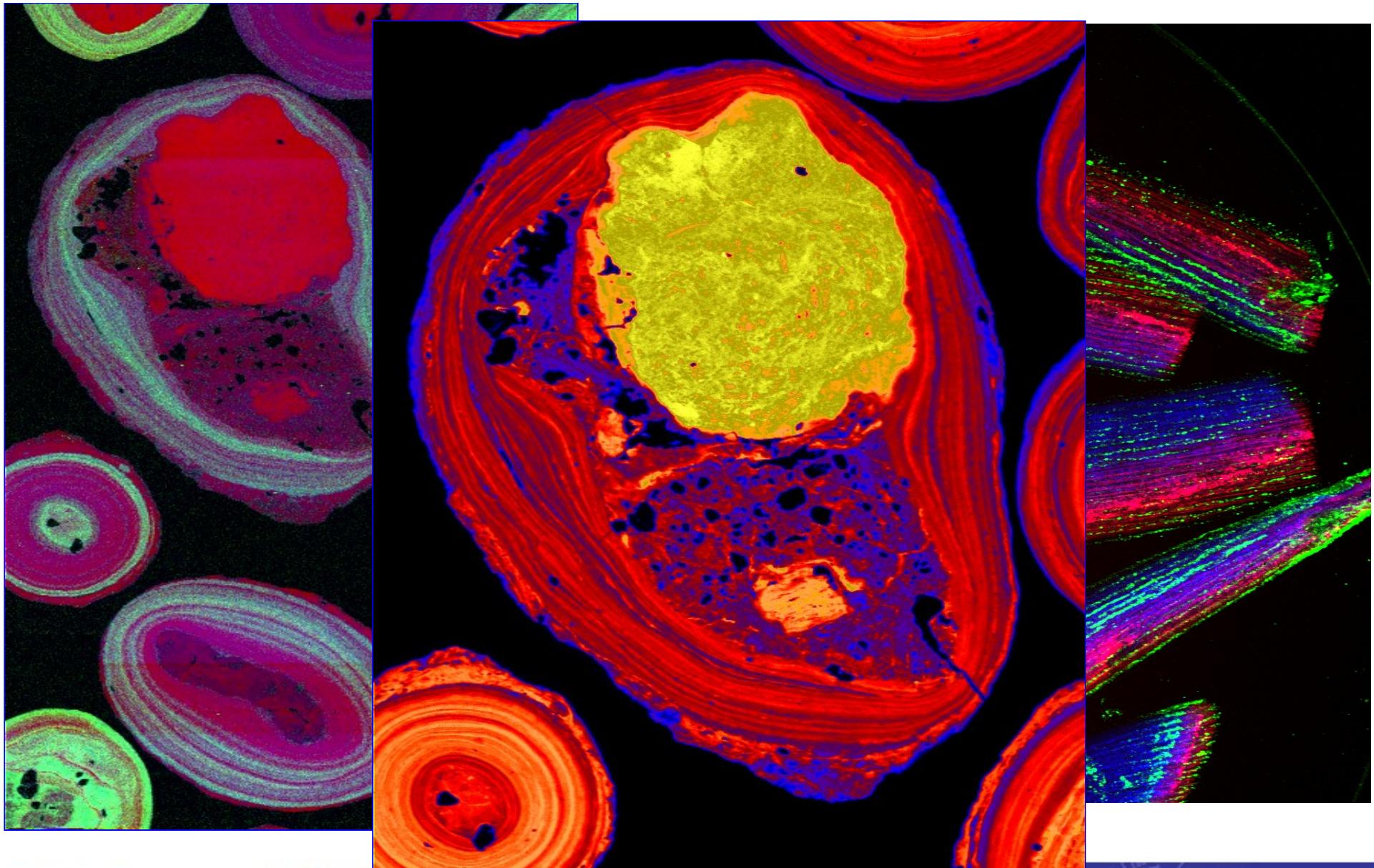
Fe-Y-Cu RGB composite (1500 x 2624 pixel images, 13 x 21 mm<sup>2</sup>)



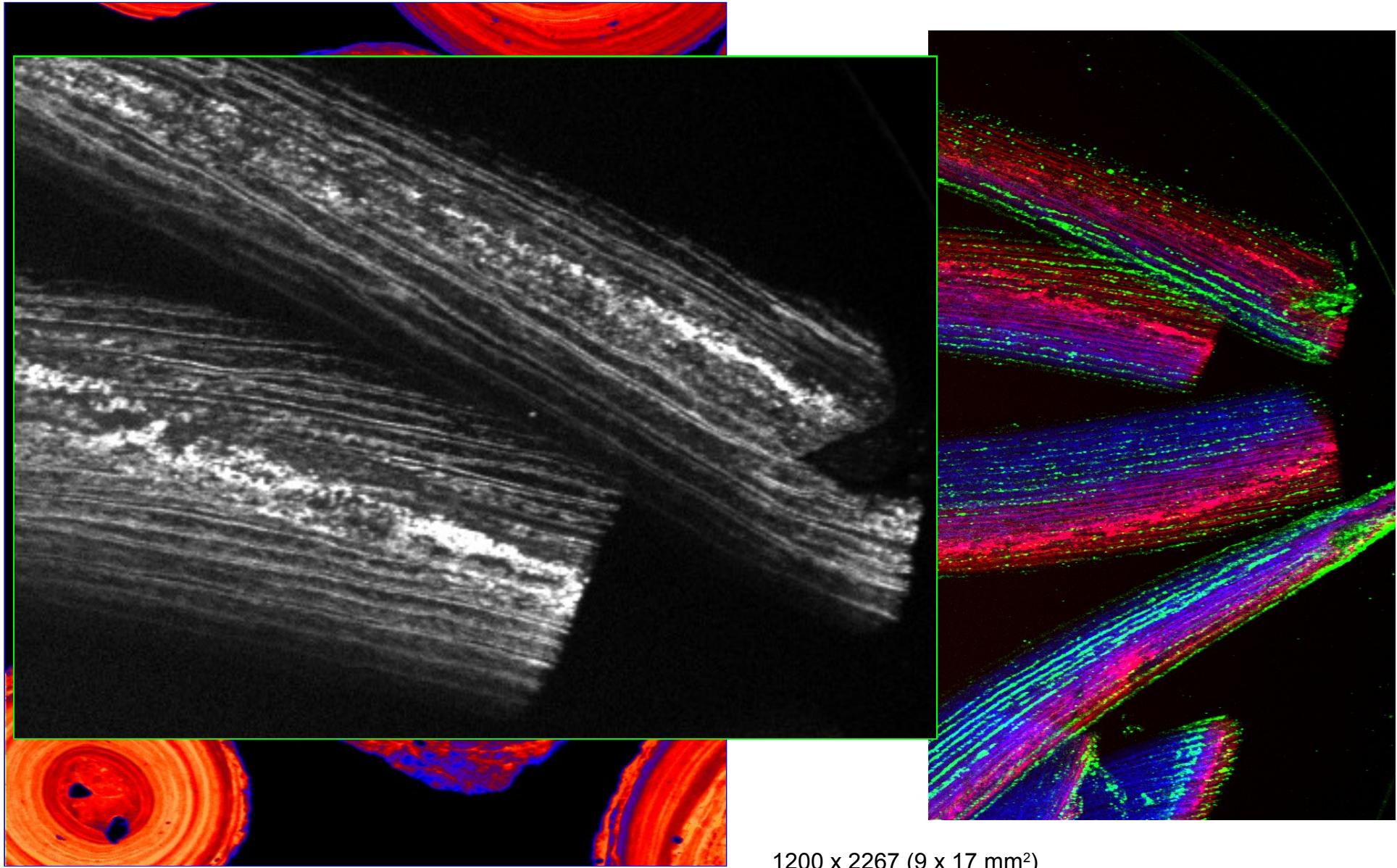
1200 x 2267 (9 x  
17 mm<sup>2</sup>)  
5.7 hours (7.5 ms  
dwell)  
7.5 x 7.5  $\mu\text{m}^2$  pixels



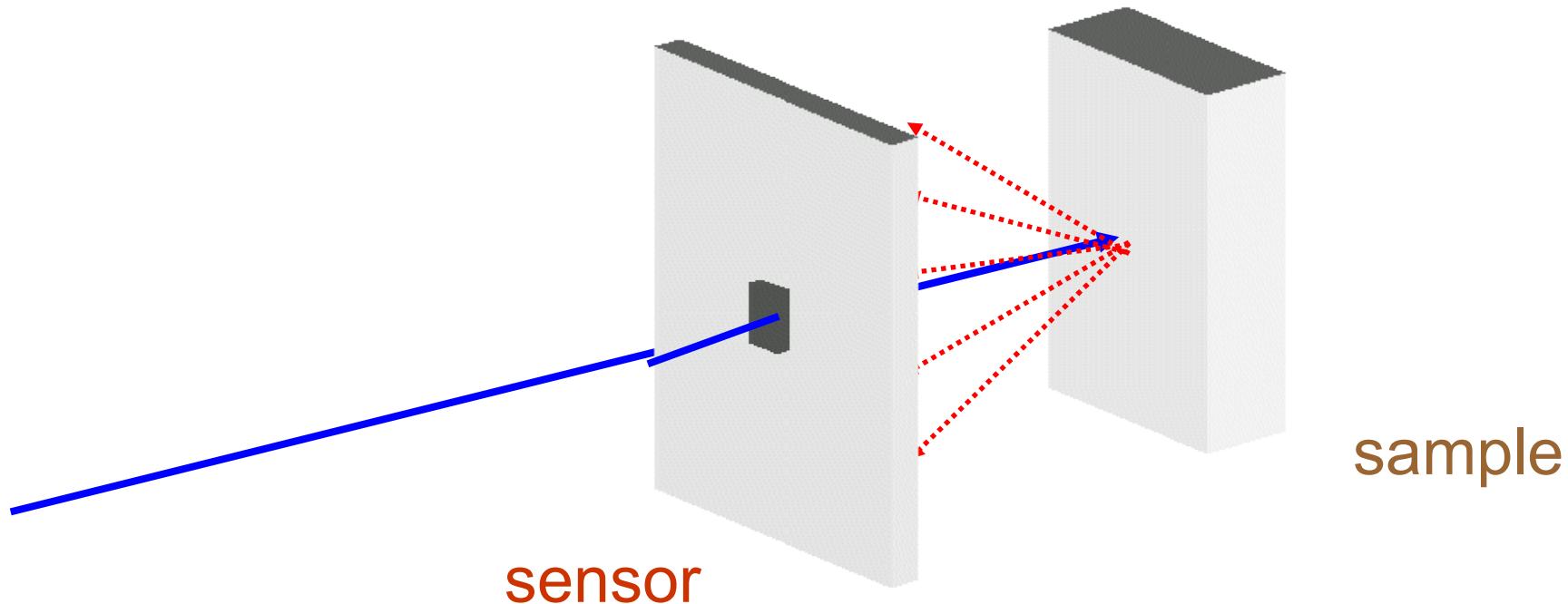
# Rapid XRF Elemental Mapping (BNL/CSIRO collaboration)



# Rapid XRF Elemental Mapping (BNL/CSIRO collaboration)



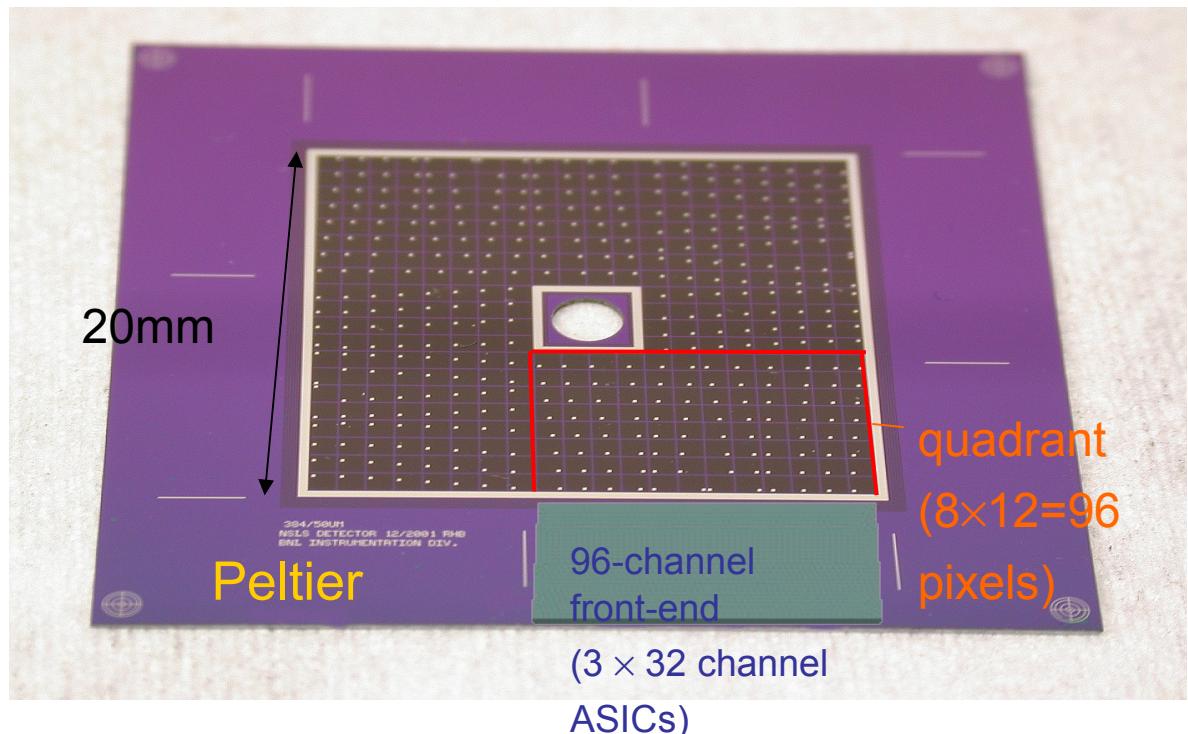
# Backscattering geometry for fluorescence microprobe



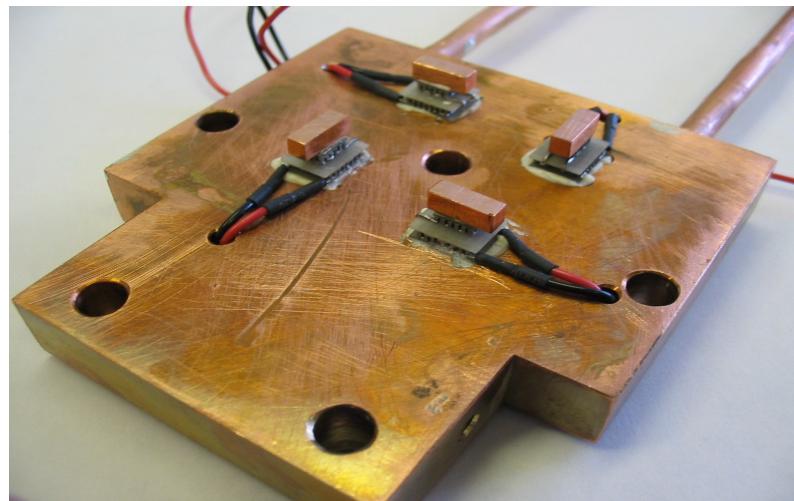
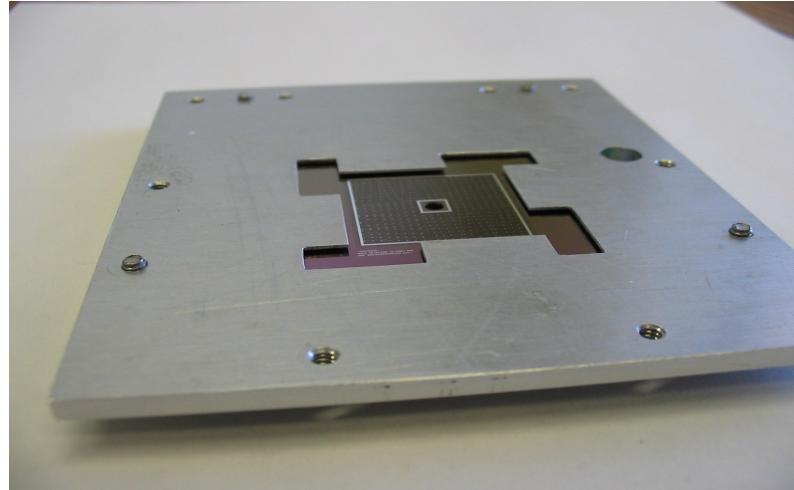
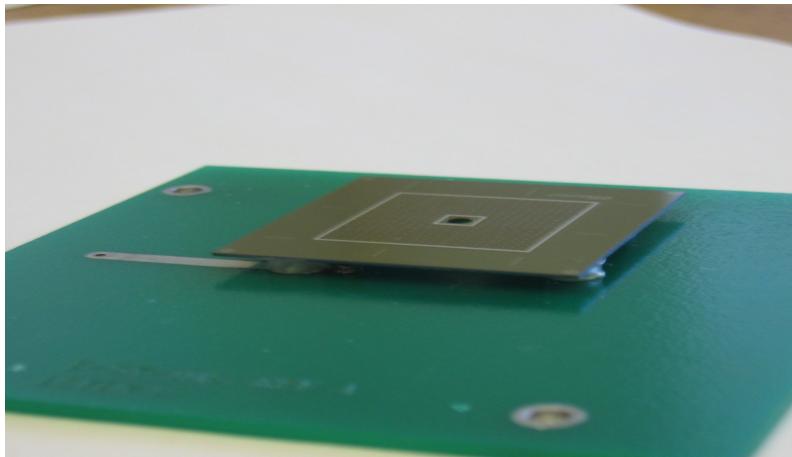
- Backscattering geometry allows close approach to sample.
- Provides good solid-angle even for small detector area

# High-rate multi-element detector for fluorescence measurements

- 384-element silicon pad array (1mm x 1mm) for absorption spectroscopy and/or x-ray microprobes.
- Central hole for incident pump beam to allow close approach to sample.
- Will use 12 BNL HERMES ASICs designed by G. De Geronimo & P. O'Connor.



# Assembly



# SRSs and FELs

- SRS is quasi-DC source (~10ns bunch spacing)

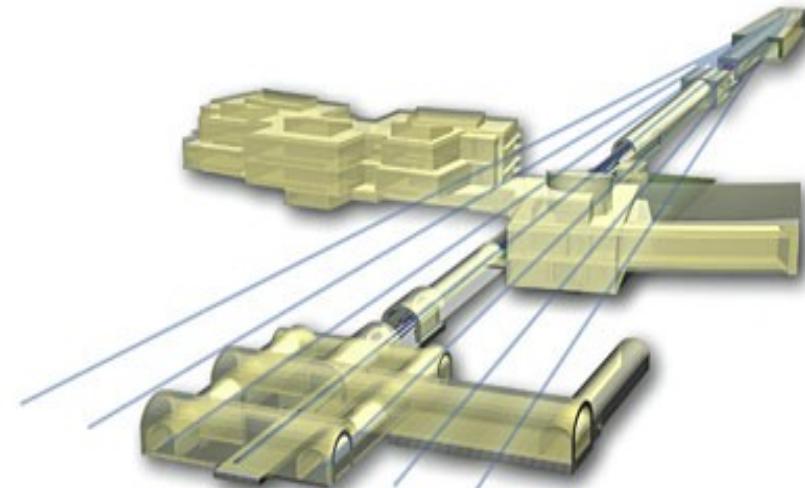
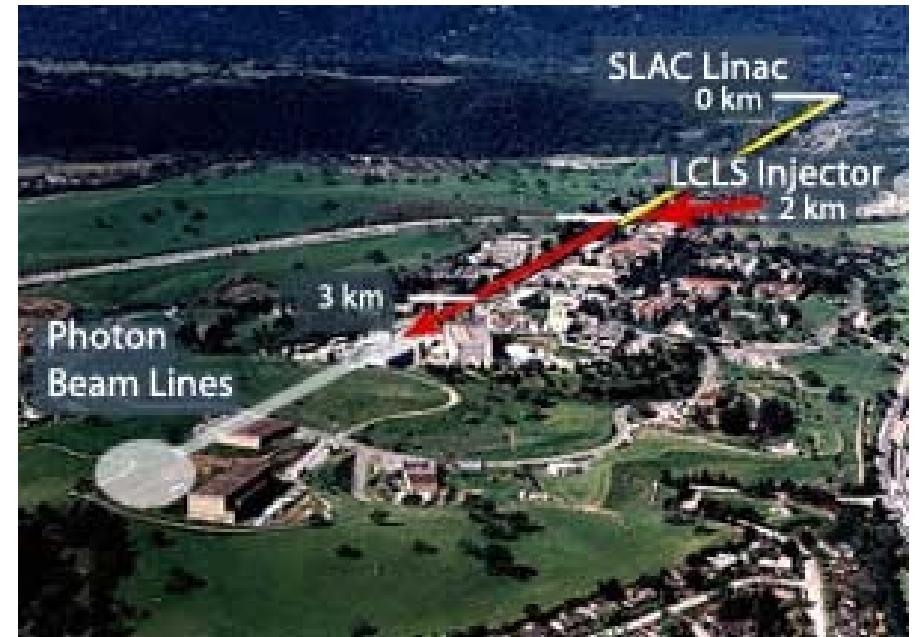
- Electron or positron storage ring
- No trigger, no 'free time' to dump data.
- High average brightness, high stability
- low peak brightness
- fairly broadband source (~1% best case without filtering)

- FEL is pulsed source (~10ms bunch spacing)

- Driven by LINAC / photocathode electron gun (low repetition rate)
- Pulse width < 1ps
- Fully transversely coherent
- Very high peak brightness
- quasi-monochromatic ( $10^{-3}$  SASE,  $10^{-4}$  Seeded)

# LCLS

- 16GeV electrons from 1/3 of SLAC
- 1.5 - 15 Angstrom radiation
- 5-6 end stations
- Operational 2009

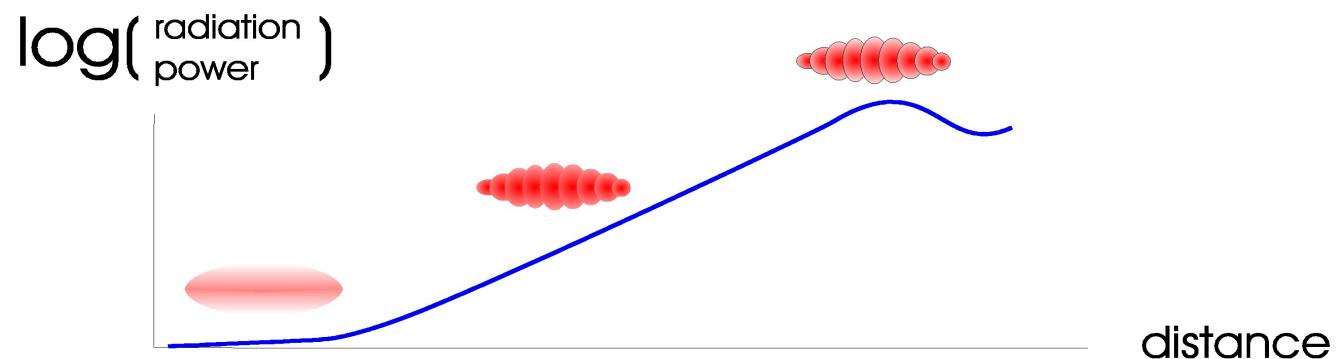
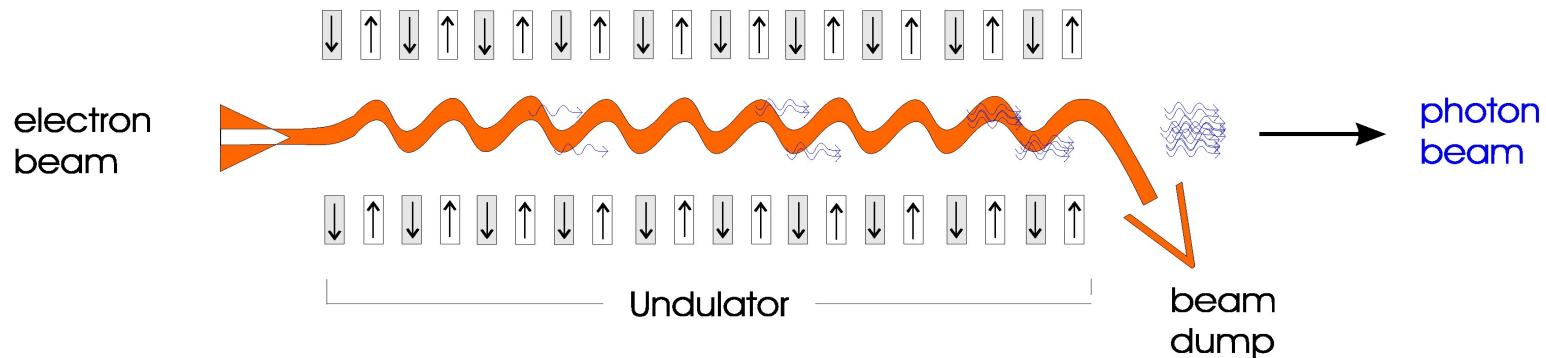


# The XFEL project (DESY)

- 20GeV LINAC
- Remote green field site for end-stations
- Very intense
- 10Hz rep. rate (~1ms macropulse with 200ns sub-period)
- Based on TESLA technology



# What is an F.E.L.?



- Undulator radiation spatially modulates electron beam
- radiation from successive microbunches is coherent
- more radiation makes deeper bunching -> more radiation

# Detectors for FELs

- No suitable commercial detectors
  - CCDs ?
  - CMOS imagers ?
- Both facilities (LCLS and XFEL) have begun a custom development
  - Specifications
- BNL development proposal to LCLS
  - Switch-matrix structure for P-P experiments
  - “Charge-pump” structure for XPCS experiments
  - Readout system
  - Data handling

# Specifications

- Source: 100fs pulses at 120Hz -> no photon counting, so need integrating detector.
- Two applications with very different specifications:
  - X-ray Pump-Probe
    - ~100% efficient @ 8keV
    - < 1 photon readout noise
    - $10^4$  photons full-well
    - ms readout time (< 8ms)
    - Extremely challenging spec:  $>10^4$  S/N, single-shot, fast readout.
  - X-ray Photon Correlation Spectroscopy
    - 100 photons full-well
    - << 1 photon readout noise, needs different technology
    - ms readout time.

# Active-matrix Area Detector

- Fully pixellated hybrid detectors (i.e. Amplifier per pixel, separate sensor array) are complicated and tend to have large pixels.
- Sensor array must be bump-bonded to CMOS circuit
  - 3 separate vendors: CMOS device, sensor array and bonder
- Monolithic devices built on fully-depleted high-resistivity silicon provide simplest structure
  - Large-area devices possible without gaps
  - No bump-bonding
  - Fully depleted wafer -> good efficiency
  - Simplest structure is monolithic active-matrix type
    - Switching mechanism integrated with sensor
    - Small pixels in principle possible (no on-pixel amps)
    - row-by-row parallel readout by off-sensor amplifiers
    - N readout channels instead of  $N \times N$ , modular readout from edge of detector by a few (~16) small ASICs
- Need to develop technology to form transistors directly on high-resistivity silicon substrate.

# XAMPS for LCLS

- Will be discussed in detail in a later talk (G. Carini, 10:50 today)

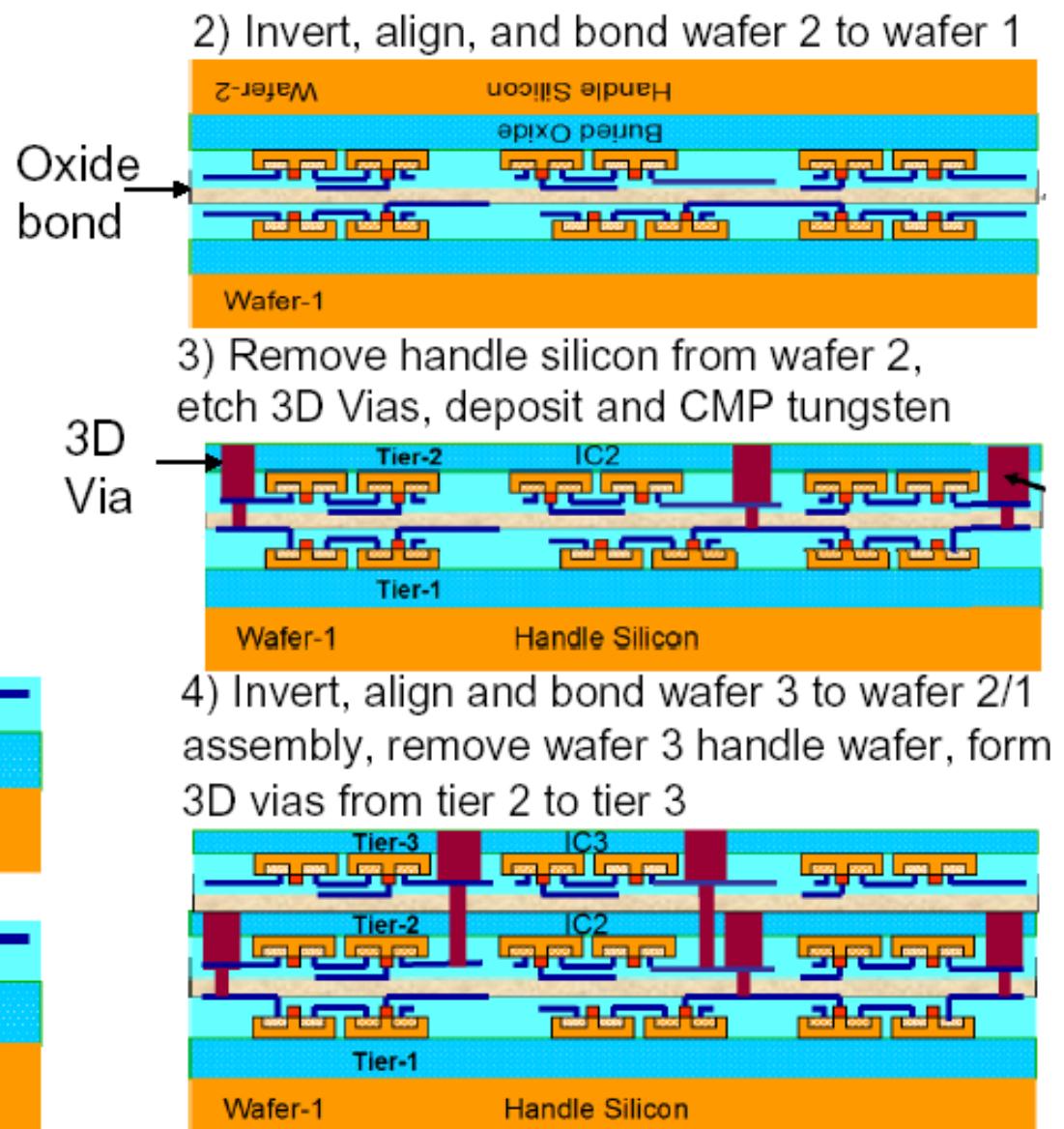
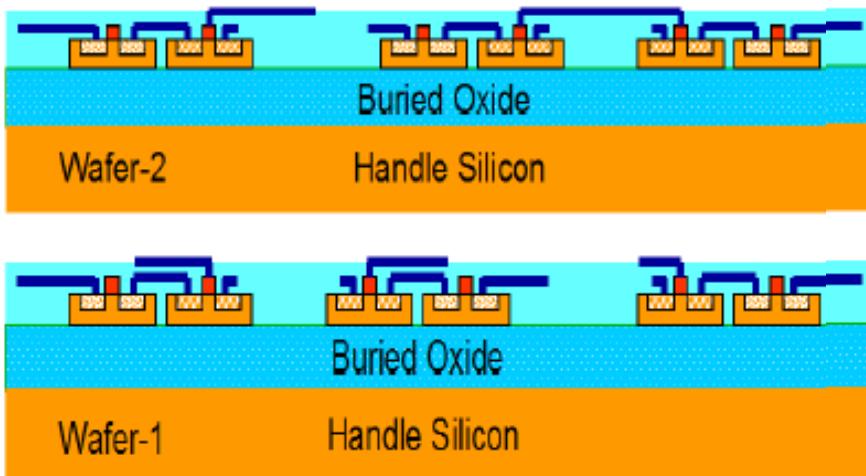
# Future developments

- Need to provide more functionality on-pixel
  - low-noise spectroscopy (<20e)
  - deep fast time framing / readout
  - time-correlation spectroscopy
- 3D integration?

# Process flow for 3D Chip

- 3 tier chip (tier 1 may be CMOS)
  - 0.18  $\mu\text{m}$  (all layers)
  - SOI simplifies via formation
- Single vendor processing

1) Fabricate individual tiers



# Summary

- SR experiments are slowly learning to use modern detector technology.
- Funding agents are slowly realizing that new sensor technologies can provide improved performance.
- New sources raise new challenges for detector developers.
- 3D integration will certainly play a role in the future.

# Collaborators

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